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DEVELOPMENT AND TESTING OF A FIVE AH SILVER-ZINC CELL

FINAL REPORT COVERING PERIOD
JULY 1967 THROUGH APRIL 1969
UNDER CONTRACT NAS 3-10924

MCDONNELL DOUGLAS ASTRONAUTICS COMPANY

MCDONNELL DOUGLAS CORPORATION

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NOTICE

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FINAL REPORT

DEVELOPMENT AND TESTING OF A FIVE AH SILVER-ZINC CELL

by

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prepared by

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ABSTRACT

Task I of this program was devoted to refining a 5 Ah silver-zinc cell design using an inorganic separator. Polysulfone material was retained for the case and cover. Electrolyte concentration was changed to 40% KOH. Pressure relief valves set at 40 psig were adapted to the cover. Gassing measurements were made on two cycling regimes at two temperatures. A slight modification of the inorganic separator cut down the gassing significantly.

Task II covered some electrical tests at high rate, wet stand tests which were satisfactory over one year at 25°C, and environmental tests (shock, acceleration, vibration) which showed no detrimental effect on the charged cells.

Task III covers the fabrication of 210 cells in 6 different lots to determine reproducibility of manufacture. The cells were divided as follows: 6 lots of 25 each were delivered to NASA and 6 lots of 10 cells were submitted to testing in our own laboratory. The cells were tested on five different cycling-temperature regimes and on charged wet stand. On the average, the cells are capable of 1500 cycles on the 1.5 hr-cycling period and 20% depth of discharge (based on 7.5 Ah original capacity). After 6-month charged wet stand, they will retain at least 80% of their capacity. With some cycling and 12-month wet life, they still deliver 20% over their rated capacity (5 Ah).

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Section 1
INTRODUCTION

The conventional silver-zinc cell is restricted in operation because of its limited cycle life. Improvement of the cycle life would qualify this energy source for many applications for which less energetic systems are now used, as in synchronous orbit satellites.

The key to improved life lies in developing a nonreactive separator to eliminate most of the factors that cause degradation and early failure.

Laboratory cells using a McDonnell Douglas inorganic separator on contract NAS 3-7639 (Reference 1) have shown improved cycle life and the ability to operate over a wide temperature range. Multiplate cells were designed and tested. Additional work was required on the present contract to refine the cell design and to extensively characterize the 5 Ah cells on various cycling regimes.

Section 2
SUMMARY

The present report covers the work performed during the 21 month period of the program to develop and test the 5 Ah silver-zinc cell designed and partially tested on a previous program (Reference 1).

The present program consisted of three tasks. Task I was a study of all cell components with particular emphasis on the sealing. 40 psig pressure relief valves were adapted to the cell and gassing rate characteristics were investigated in free-venting and pressurized cells on three different cycling period and temperature combinations. Task II was concerned with the preliminary cell testing of the approved design incorporating all improvement features of the previous task. Charged wet stand tests were run at 100°C and 25°C. Capacity retention was 80% after a 6-month charged stand at 25°C. Task III covered the fabrication test and delivery of several cells made in six different lots to determine reproducibility of manufacture. Sixty cells were retained and tested in our laboratory as five different cycling-temperature regimes. One hundred and fifty cells were delivered to Crane Laboratory for tests as instructed by NASA.

Specifications of the 5 Ah cell may be found in Appendix B.

Section 3
WORK PERFORMED

Work done on Contract NAS 3-7639 (Reference 1) has shown that a silver-zinc cell designed with the Douglas inorganic separator 3420-09 (rigid type) is capable of improved cycle life at temperatures as high as $100^{o}C$.

The present program requires that a refined 5-Ah cell design be provided suitable for extensive characterization. After a period of cell component improvement and final design selection, a number of identical cells of the approved design were delivered to NASA for independent testing in six lots of 25 cells each, fabricated at short intervals.

3.1 TASK I: SELECTION OF IMPROVED CELL COMPONENTS

The objective of this task was to improve various cell components and fabrication techniques and to incorporate these improvements into a 5-Ah cell design. The gassing characteristics were also to be investigated to provide information for the design of a fully sealed cell.

3.1.1 Electrodes

The positive and negative electrodes were essentially the same design as used on previous contract NAS 3-7639 (Reference 1). Minor modifications were tried on the negative and positive electrodes, but were not used on the majority of the tests.

3.1.1.1 Negative Electrode

The addition of 1% PbO to the zinc oxide mix of the negative electrode was tried once in a few test cells on contract NAS 3-8513 "Improvement of the Zinc Electrode" (Reference 2). This modification of the standard electrode was tested again on full 5-Ah cells. Five standard 5-Ah cells and five cells using PbO additive were built and placed on test on the 1/2-hour discharge, 1-hour charge cycling regime at 20 mA/cm^2 at room temperature. Table I and Figure 1 show their original electrical performance. Table II gives their end discharge voltages at various cycles. Figure 2 and Table II give their failure cycles and failure analyses. Figures 3 to 5 show comparative typical cycling performance at various cycles.

Two control cells failed after 2212 cycles, one cell with PbO failed after 2554 cycles, but upon dissection of the three cells, it was found that the negative electrode wire leads were loose in the terminal with solder that was heavily corroded (these early cells used feed-through terminals). Two other cells (one control, one with PbO) failed to deliver their cycling capacity at cycles 2554 and 2720, respectively. The terminals showed the same corrosion signs and were tentatively crimped around the wires for better contact. After recharge, the cells were put back on cycling. However, the first one could not cycle more than 2575 cycles, whereas the other continued cycling. The cycle life ranged from 2212 cycles to 3194 cycles. Analysis of the data shows marginal differences that may be attributable to normal statistical distribution.

The cause of failure is primarily low capacity, close to the cycling capacity requirement (Table III), which makes the cell operate at 100% of the available capacity. However, when the cell stood in the charged condition, there was a slight decay of its OCV, which was also evidence of a slow short. Upon dissection, it was noticed that the separators were not cracked in many instances, the edge seals were good, and the electrodes were in good condition. The zinc electrodes did not slump nor lose shape for more than 10 to 15% at their top. However, some separators showed signs of zinc penetration.

3.1.1.2 Shifting Positive Electrodes and Their Correction

<u>Cells with Large Positive Electrodes</u>

It was noticed that the silver electrodes may shift when cells are handled dry. In order to circumvent this undesirable shifting, the silver electrodes were made larger so as to occupy the full width of the case and touch the bottom.

TABLE I

CELLS WITH AND WITHOUT PbO ADDITIVE

(All cells charged at 0.350 A to 2.10 V
and discharged at 1.0A to 1.0V)

	Input	Output	Plateau Voltage
Control Cells			
ZL-11-1	8.4 Ah	7.3 Ah	1.42 V
ZL-11-2	8.4	7.2	1.42
ZL-11-3	8.4	7.4	1.42
ZL-11-4	8.4	7.2	1.42
ZL-11-5	8.4	7.3	1.42
Average	8.4 Ah	7.3 Ah	1.42 V
Cells with PbO			
ZL-11-6	10.7 Ah	8.6 Ah	1.43 V
ZL-11-7	10.7	8.7	1.43
ZL-11-8	10.7	8.5	1.43
ZL-11-9	10.7	8.5	1.43
ZL-11-10	10.7	8.5	1.43
Average	10.7 Ah	8.6 Ah	1.43 V

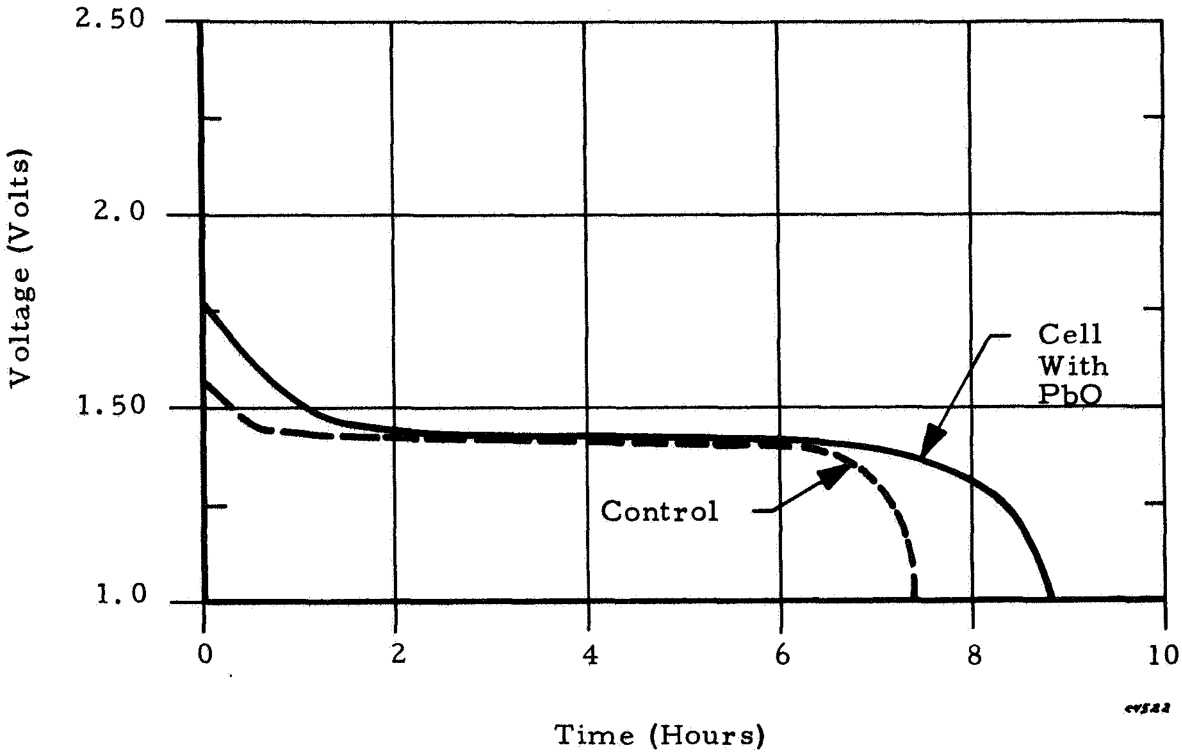

Figure 1. Discharge Curves at 1 A
(Cell Specs as Indicated)

TABLE II

VOLTAGE AT END OF DISCHARGE PERIOD (1/2 HOUR)

Regime: 1/2 hr – 2.5 A discharge
1 hr – 1.4 A charge

Design Feature	Cell No.	Cycles									
		5	500	1000	1500	1930	2250	2500	2800	2900	3194
Standard Negative	ZL-11-1	1.40	1.30	1.28	1.28	1.34	1.29	1.29	1.29	–	–
	ZL-11-2	1.40	1.34	1.34	1.34	1.36	1.31	1.28	1.28	–	–
	ZL-11-3	1.40	1.31	1.30	1.30	1.32	–	–	–	–	–
	ZL-11-4	1.40	1.30	1.28	1.28	1.32	1.32	1.30	1.29	–	–
	ZL-11-5	1.40	1.28	1.28	1.28	1.32	–	–	–	–	–
	Average	1.40	1.31	1.30	1.30	1.35	1.31	1.29	1.29	–	–
PbO in Negative	ZL-11-6	1.40	1.30	1.28	1.26	1.30	1.26	1.30	1.26	–	–
	ZL-11-7	1.40	1.32	1.30	1.28	1.34	1.30	1.30	1.30	1.18	–
	ZL-11-8	1.40	1.31	1.28	1.27	1.30	1.30	1.30	–	–	–
	ZL-11-9	1.40	1.34	1.34	1.34	1.35	1.36	1.20	1.20	1.22	0.98
	ZL-11-10	1.40	1.30	1.27	1.26	1.24	1.20	1.20	1.20	–	–
	Average	1.40	1.31	1.29	1.28	1.30	1.28	1.26	1.25	1.20	0.98

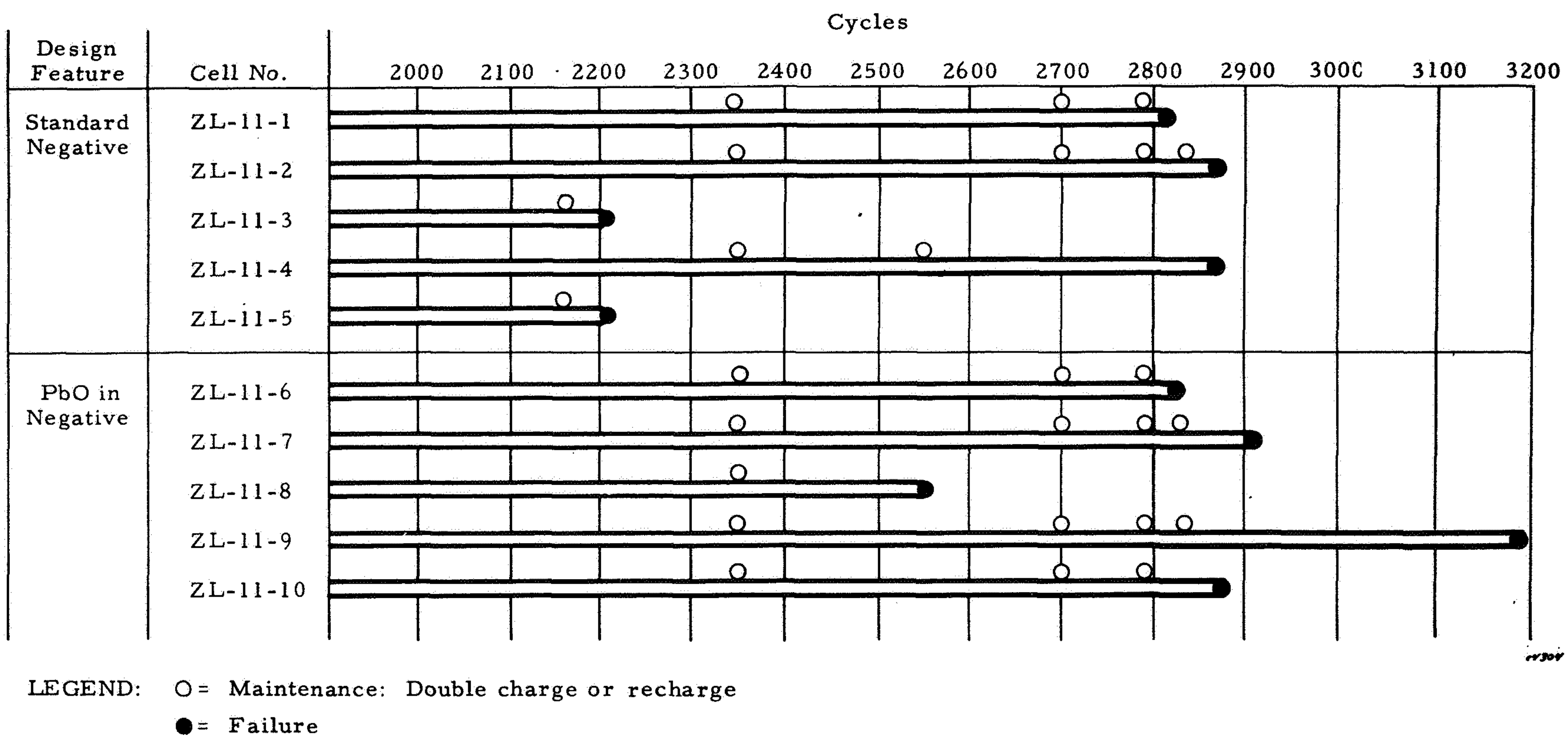

Figure 2. Cycling Performance of Cells on the 90 Minute Regime

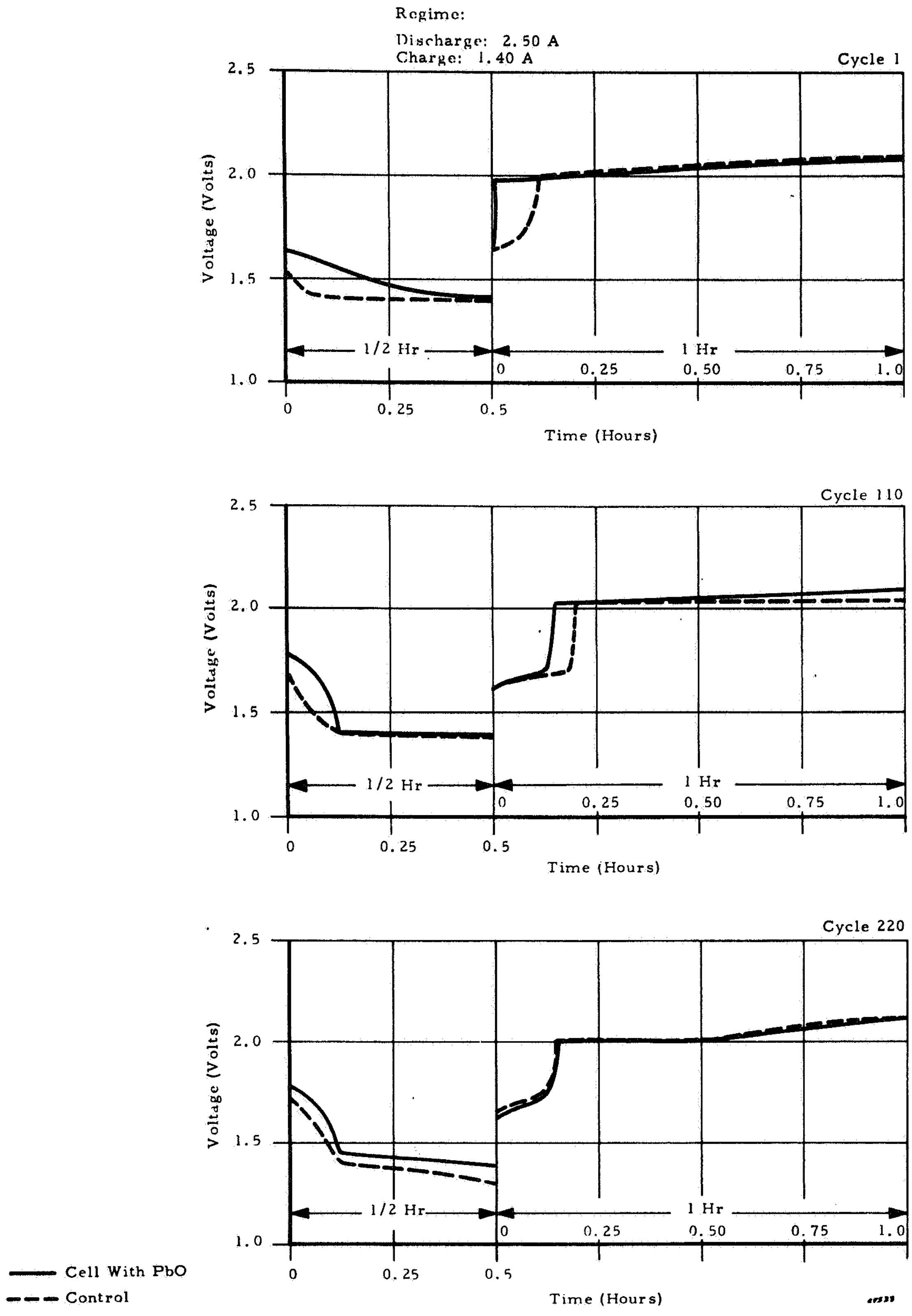

Figure 3. Cycling Curves of Cells With and Without PbO

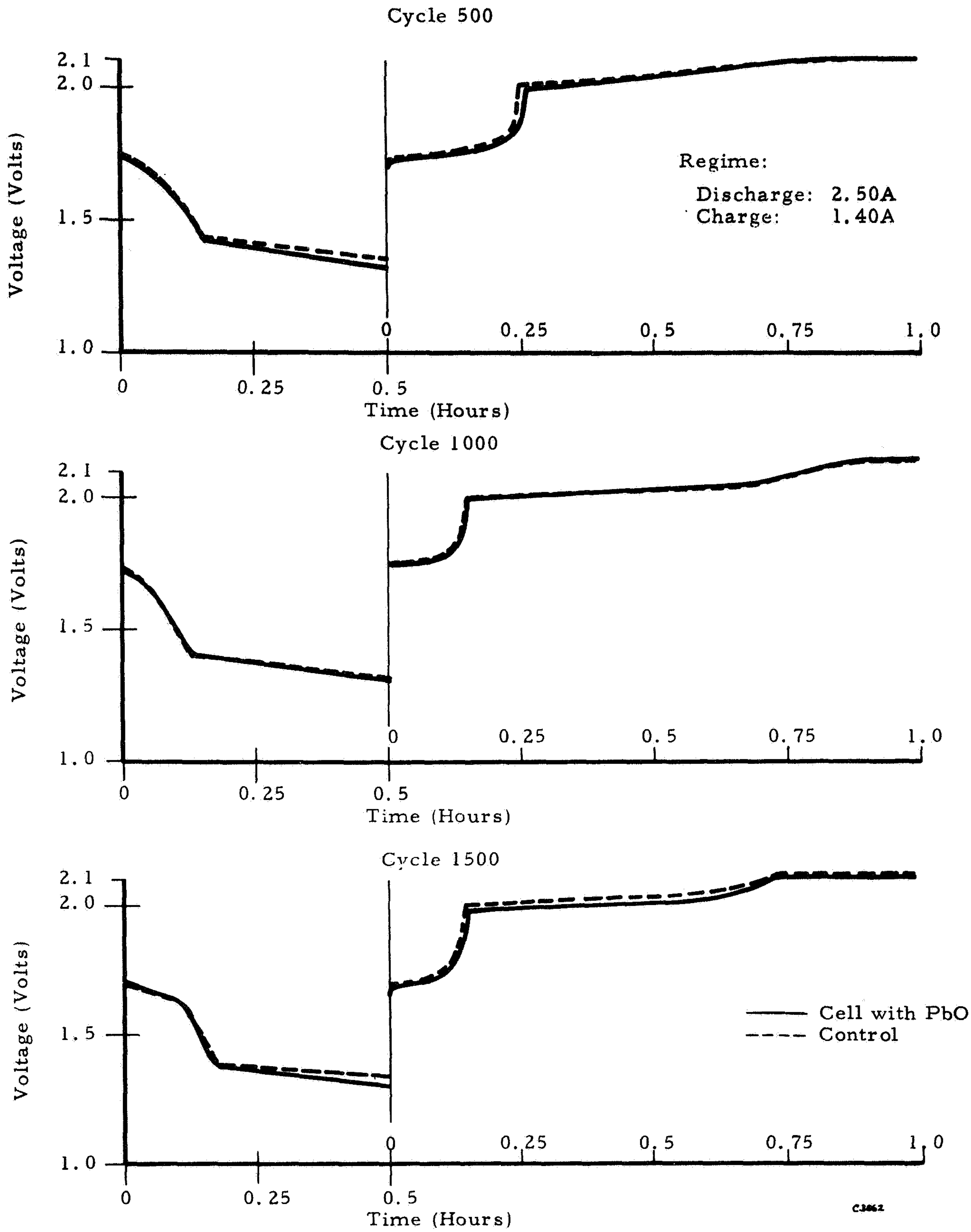

Figure 4. Cycling Curves of Cells With and Without PbO

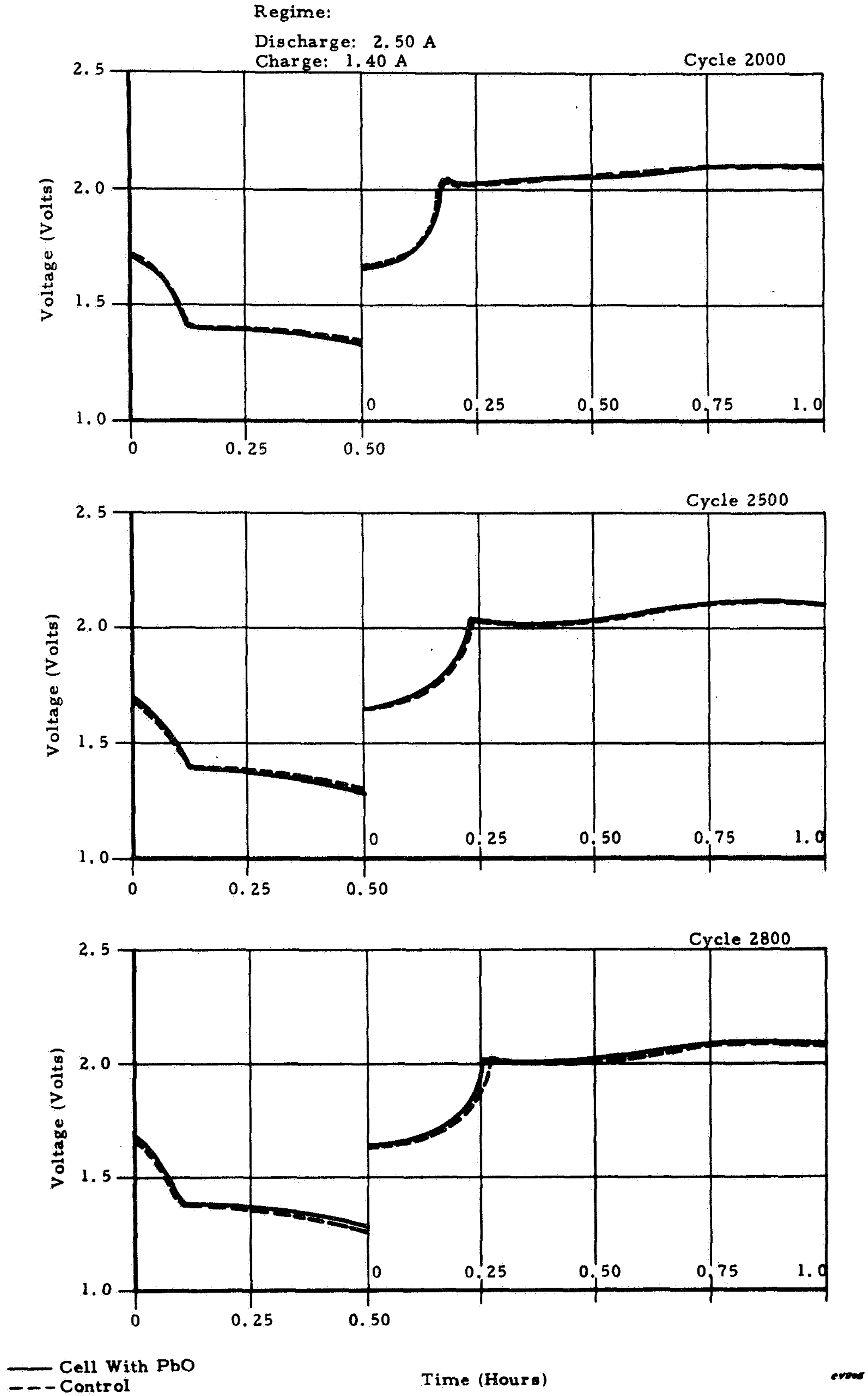

Figure 5. Cycling Curves of Cells With and Without PbO

TABLE III

FAILURE ANALYSIS OF ZL-11 SERIES

Cell No.		Capacity at			OCV Stand		
Standard	Failure Cycle	2.5 A	Drain at 0.5A	Total	0 Hr	2 Hr	Examination
ZL-11-1	2809	1.70 Ah	0.35 Ah	2.05 Ah	1.82 V	1.78 V	No cracks
ZL-11-2	2783	2.00 Ah	0.25 Ah	2.25 Ah	1.83 V	1.80 V	No cracks
ZL-11-3	2212	—	—	—	—	—	One slight
ZL-11-4	2873	0.8 Ah	0.25 Ah	1.05 Ah	1.82 V	1.80 V	One severe
ZL-11-5	2212	—	—	—	—	—	No cracks
Additive							
ZL-11-6	2848	1.25 Ah	0.35 Ah	1.60 Ah	1.82 V	1.76 V	No cracks
ZL-11-7	2905	1.90 Ah	0.15 Ah	2.05 Ah	1.84 V	1.81 V	No cracks
ZL-11-8	2554	—	—	—	—	—	One slight
ZL-11-9	3194	0.25 Ah	0.25 Ah	0.50 Ah	1.81 V	1.78 V	Four severe
ZL-11-10	2894	1.90 Ah	1.00 Ah	2.90 Ah	1.84 V	1.83 V	No cracks

The cost of the extra silver is negligible compared with the labor involved in building plastic frames around each electrode.

Five cells were built and compared with five controls. Table IV and Figure 6 show the differences between the two groups of cells in formation capacity and discharge voltage.

The cells were then placed on automatic cycling on the 90-minute period regime (2.5 A discharge for 1/2 hour, 1.4 A charge for 1 hour). The group of cells with large electrodes exhibited erratic behavior and were discontinued after 100, 145 and 300 cycles for rebalancing individual cells. After 432 cycles, they were finally discontinued, whereas the control reached 500 cycles without adjustment.

The excess silver may have created, among other factors, a zinc limiting condition faster than in the regular cells. It was decided to drop this approach and use another method for immobilizing the silver electrodes in the control cells.

Immobilized Cell Pack

It was found more expedient to pour a thin layer of epoxy (3 grams) in the bottom of the cell case before inserting the cell pack. After cure, the epoxy immobilizes the cell pack in all directions without getting into the electrodes or the separators because of its relatively high viscosity.

3.1.2 Separator System

3.1.2.1 Separators

Type 3420-09 is used exclusively. The specifications as established on previous contracts (NAS 3-7630 (Reference 1) and NAS 3-8513 (Reference 2)) were maintained throughout this program.

All separators were 100% inspected and segregated for exclusive use on this task. The detailed order was as follows:

TABLE IV

CAPACITY ON FORMATION

Positive	Cell No.	Output	Plateau Voltage
1.6" x 1.6" (Control)	ZL-24-1	7.85 Ah	1.46 V
	ZL-24-2	7.90 Ah	1.46 V
	ZL-24-3	7.75 Ah	1.45 V
	ZL-24-4	8.10 Ah	1.45 V
	ZL-24-5	8.10 Ah	1.45 V
	Average	7.90 Ah	1.45 V
1.88" x 1.95" (Large)	ZL-24-6	9.50 Ah	1.44 V
	ZL-24-7	9.60 Ah	1.44 V
	ZL-24-8	9.60 Ah	1.44 V
	ZL-24-9	9.60 Ah	1.43 V
	ZL-24-10	8.60 Ah	1.44 V
	Average	9.40 Ah	1.44 V

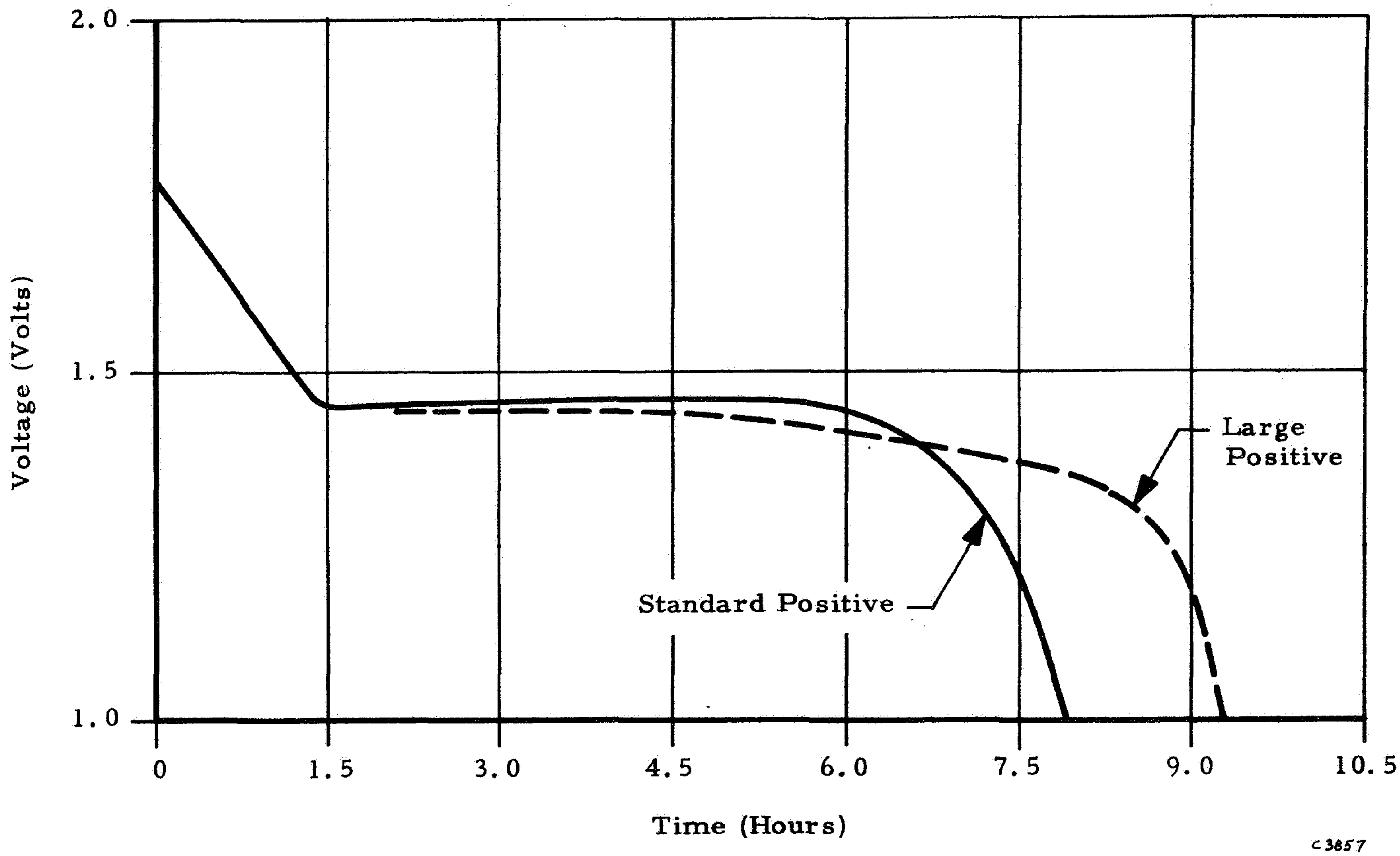

Figure 6. Discharge of Curves of Cells With Two Different Sizes of Positive Electrodes

1. Absorption: 9.5-10.5% (run absorption check on all pieces)
2. Thickness: 0.025" ± 0.001"
3. Must be flat
4. Must be free of holes, chips, discolorations, cracks, pits, and other mechanical imperfections.
5. All pieces must be examined under at least a 10X microscope to be certain there are no microfissures, cracks, etc.

For this work, a new batch of granulated pressing powder is made and its identity is kept separate throughout the manufacturing process.

3.1.2.2 KT Interseparators

Because of the discontinuance of the present KT paper by the supplier, a new KT made by another supplier was acquired and compared with the previous one in the presence of KOH at 25°C and at 100°C. Samples soaked in excess KOH, using two different concentrations (30 and 45%), were left on stand up to 75 days along with blanks (KOH without materials) to establish the variations of free KOH content and carbonation. The liquors were analyzed initially, after 20 and 75 days. For a given set of conditions, temperature and KOH concentration, the three liquors show no significant difference within the experimental errors (Table V).

3.1.3 <u>Case</u>

Cases made of polysulfone P-1700 and polyphenylene oxide 534-801 were used for case-to-cover seal investigation and ultimate material selection (see Paragraph 3.1.4). However, in the absence of any obvious disadvantage for either material, polysulfone was constantly used because of its transparency which permits easier quality control in the final assembly stage.

3.1.4 <u>Case-to-Cover Seal</u>

As a preliminary testing procedure for seal integrity, it was decided to pressure test each case with dry nitrogen at 100 psig for 5 minutes at 25°C. A test fixture was designed and built for this purpose. Figure 7 is a schematic of the set up and the detailed testing procedure. Two sealing methods were investigated.

TABLE V

EFFECT OF KOH ON KT INTERSEPARATOR MEASURED BY VARIATION OF CONTENTS (g/ℓ) OF FREE KOH AND CARBONATE

Material in KOH	Temperature	30% Solution						45% Solution					
		Free KOH			Carbonate			Free KOH			Carbonate		
		Initial	20 Days	75 Days	Initial	20 Days	75 Days	Initial	20 Days	75 Days	Initial	20 Days	75 Days
Blank	25°	382	387	379	21	12	38	647	666	607	17	23	35
KT-Original	25°C		378	360		19	41		657	599		21	45
KT-New			389	353		17	31		642	605		23	38
Blank	100°C	384	384	407	17	16	31	636	655	671	17	19	35
KT-Original			400	381		21	21		653	641		23	38
KT-New			404	393		19	21		652	609		21	31

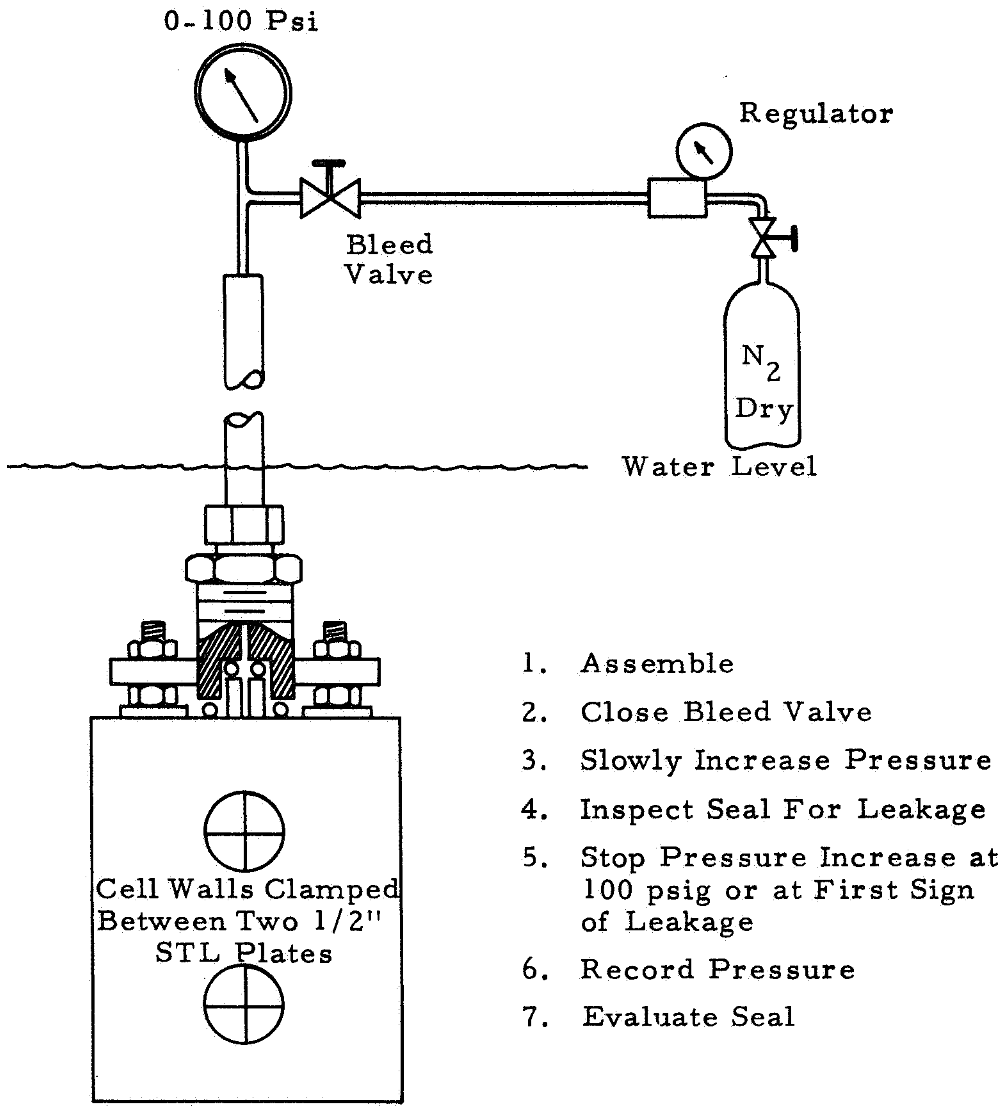

Figure 7. Text Fixture for Cast-to-Cover Seal

3.1.4.1 Hot-Gas Welding

This was done by using a "Corofac" Model EHGG-4000 welding gun and accessories. The welding is made with round filler rods of parent materials (1/8" diameter). Preliminary sealing experiments were started on polysulfone cases to get an idea of the variability inherent in the method and of the possible parameters, such as time, annealing, design configuration.

Using available 1/8" diameter polysulfone welding rods and keeping the gas pressure constant at 6 psi, the temperature was determined as a function of the distance from gun tip to weld area. Table VI is a quick evaluation on a few cases.

TABLE VI

HOT-GAS WELDING TEST

Case	Distance	Temperature	Remarks
ZL-7-1	1/8"	463°F	Scorched; bad
ZL-7-2	1/4"	387°F	Scorched; bad
ZL-7-3	1/2"	386°F	Scorched; bad
ZL-7-4	3/4"	320°F	No scorching; good
ZL-7-5	3/4"	324°F	No scorching; good
ZL-7-6	1"	332°F	Fair

During this test, the thin wall at the top of the case (1/16" thick) distorts and melts before the 1/8" rod does. Attempts to procure 1/16" rods were unsuccessful (either not available or too expensive to get them extruded). When the concurrent investigation of another welding method (ultrasonic welding) showed more promising results, no additional work was attempted on the hot gas welding.

3.1.4.2 Ultrasonic Welding

A Branson J-32 Ultrasonic Welding System was used with a specially designed titanium horn. Tests on PS (polysulfone) and PPO (polyphenylene oxide) cases were started to establish the optimum values for proper sealing of the following parameters:

1. pressure of the horn
2. weld time
3. hold time, after weld
4. power

The object was to develop an operating procedure that will produce a seal capable of withstanding an internal pressure of 100 psig. As a start, it must meet the requirement of withstanding 100 psig with no drop in pressure or no bubbles under water for 5 minutes.

Annealing

PS and PPO cases were annealed before welding. The most suitable annealing procedure found is the following:

Material	Medium	Temperature	Part	Time
Polysulfone	Glycerine	330°F	Case	2 minutes
			Cover	4 minutes
PPO	Glycerine	295°F	Case	1 hour
			Cover	1 hour

After this procedure, all internal stresses were eliminated. When stresses are present, the parts craze or crack when in contact with acetone (5 seconds for PS material) and P-Dioxane (5 minutes for PPO material). After this procedure, the parts submitted to the same organic liquids for the same periods of time did not experience any failure (craze or crack).

Welding

The parameters involved were optimized and held to the following:

Material	Pressure	Weld Time	Hold Time
PS	92 psi	2.5 sec.	1.0 sec.
PPO	92 psi	2.75 sec.	1.5 sec.

Eight PS cases and five PPO cases consecutively welded passed the pressure test (100 psi for 5 minutes at room temperature) one after the other without leaking.

To go one step further, several welded cases were prepared for testing for 200 hours with a pressure differential of 50 psig at 25°, 100°, and 135°C.

As a sideline, an attempt was made to determine the water vapor permeability constant, P, for polysulfone and PPO at various temperatures by sealing a certain amount of 45% KOH in welded and top-potted cases. One case of each material was submitted for 120 hours to each of the following temperatures: 25°C, 50°C, 75°C and 135°C. A control case without KOH was also tested at 135°C. The cases were accurately weighed before and after the test and checked for alkali traces. No alkali trace could be detected after the test along the seals, even after inverting the cases for 64 hours so that KOH was in direct contact with the seals.

Table VII is a list of the data. When plotted on a semi-logarithmic graph, the data fit remarkably a straight line (Figure 8) which shows that the weight loss being an exponential function of the temperature may be due solely to the diffusion of gases through the case plastic. This offers further evidence that leakage through poor sealing can be ruled out.

TABLE VII

WATER WEIGHT LOSS THROUGH SEALED CASES AFTER 120 HOURS (GRAMS)

Material	Temperature	Original Weight	Final Weight	Difference	Remarks
Polysulfone P-1700	50°C (KOH)	76.795 g	76.721 g	0.074 g	
	75°C (KOH)	77.350	77.192	0.157	
	100°C (KOH)	77.030	76.747	0.283	
	135°C (KOH)	76.564	75.664	0.900	Crazing
	135°C (dry)	62.983	62.854	0.129	
PPO (534-801)	50°C (KOH)	69.033	68.982	0.051	
	75°C (KOH)	71.443	71.325	0.118	
	100°C (KOH)	69.914	69.654	0.260	
	135°C (KOH)	70.889	70.070	0.819	Turned Darker
	135°C (dry)	55.719	55.665	0.054	Turned Darker

Ultrasonically welded, fully sealed cases containing KOH appear therefore able to withstand sterilization without leaking alkali. The weight loss observed can be traceable to water vapor diffusion through the case plastic.

Evidence of the integrity of the ultrasonic weld of the cover to the case was more firmly established by repeated and varied testing at 135°C.

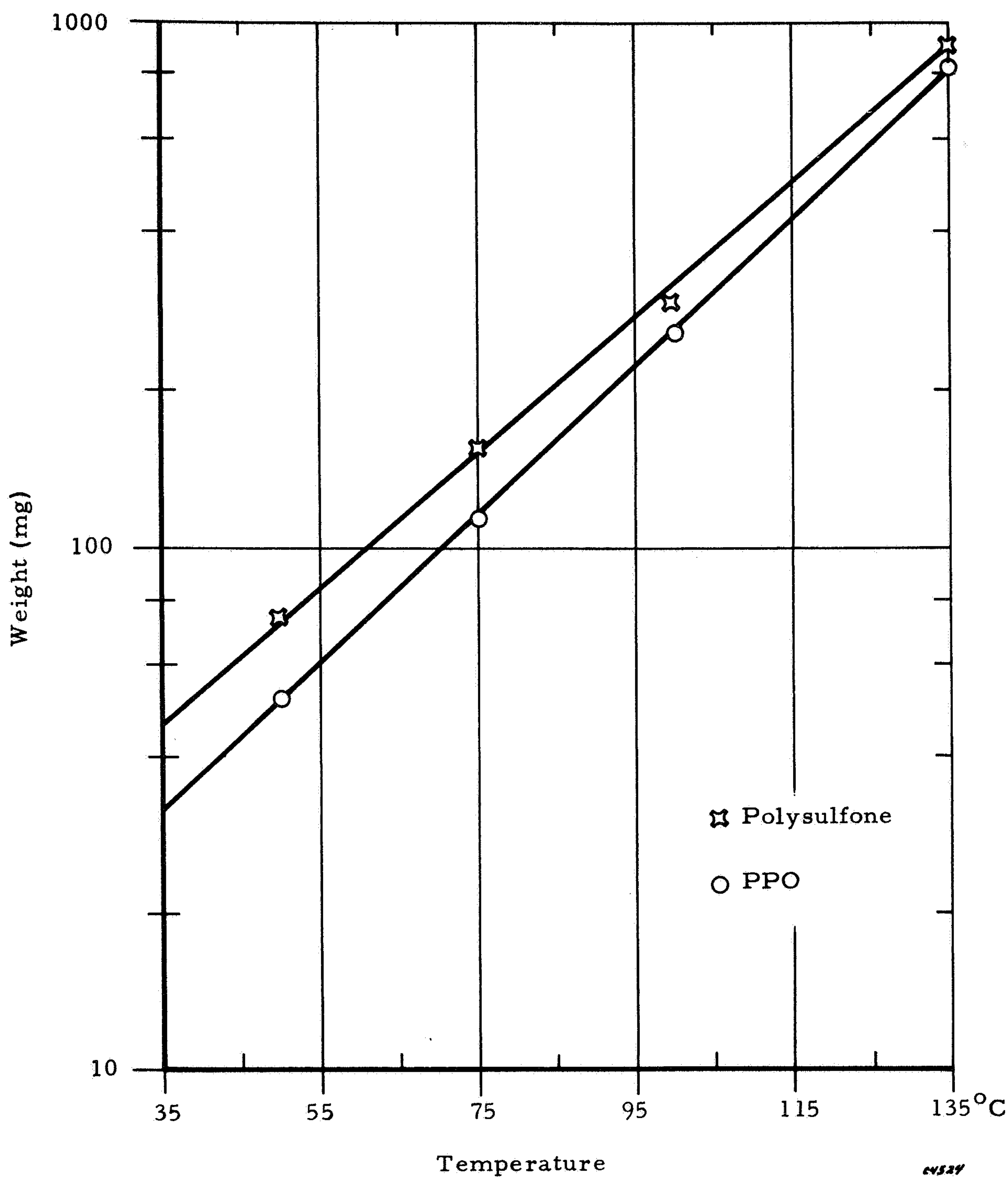

Figure 8. Water Weight Loss After 120 Hours Through Plastic 5 AH-Cell Case

Cases Partially Filled with KOH and Sealed — Five polysulfone cases and five PPO cases were filled with 10 cm^3 of 45% KOH and then sealed. Three of each group were left at $135^{\circ}C$ for 120 hours, and two of each group for 200 hours. After determining that no trace of alkali could be detected at the welded seams immediately after test, nor after 60 hours in an inverted position, their weight loss was measured. The data presented in Table VIII show less weight loss with PPO than PS over 200 hours, but in both materials the water permeability is relatively low and will be certainly tolerable to a cell undergoing a loss of less than 5% water.

Cases Pressurized at 50 psig — Three PS cases were sealed and subjected to 50 psig continuously for 200 hours at $135^{\circ}C$. Two cases passed the test successfully. The third one presented a leak after 187 hours, only at the pressure fixture adapted on the top of the cover and not at the welded seams.

Ultrasonically welded PPO cases were also submitted to the same test. A pressure drop of about 8 psig was observed during this test period which may have been due to diffusion as no leakage under water could be detected before or after the high temperature 200-hour pressure test. From these data, the calculated diffusion rate is less than 5×10^{-5} cm^3/second.

Figures 9 and 10 show the set-ups for ultrasonic welding and pressure testing of welded cases.

3.1.5 Terminals

No work was contemplated on the terminal seal. The only modification introduced was to the attachment of leads to the terminal. Instead of a hollow terminal where the leads are fed through and soldered through the top, the terminal used was a blind terminal, with a hole through the bottom about halfway up. The leads were inserted and soldered through the bottom (Figure 11). This improvement prevents any possibility of leakage through the terminal hole caused by poor soldering or solder corrosion.

Another improvement was the use of indium solder which is more resistant to alkali attack and high temperature.

TABLE VIII

WEIGHT LOSS OF SEALED CASES PARTIALLY FILLED WITH KOH AND SUBMITTED TO 135°C FOR INDICATED TIME

	120 Hours		200 Hours	
	Case	Loss	Case	Loss
Polysulfone	ZL-16-13	1.041 g	ZL-16-16	1.033 g
	ZL-16-14	0.542	ZL-16-17	1.581
	ZL-16-15	0.718		
	Average	0.800 g	Average	1.307 g
PPO	ZL-16-18	0.702 g	ZL-16-21	0.879
	ZL-16-19	0.738	ZL-16-22	0.889
	ZL-16-20	0.701		
	Average	0.740 g	Average	0.884 g

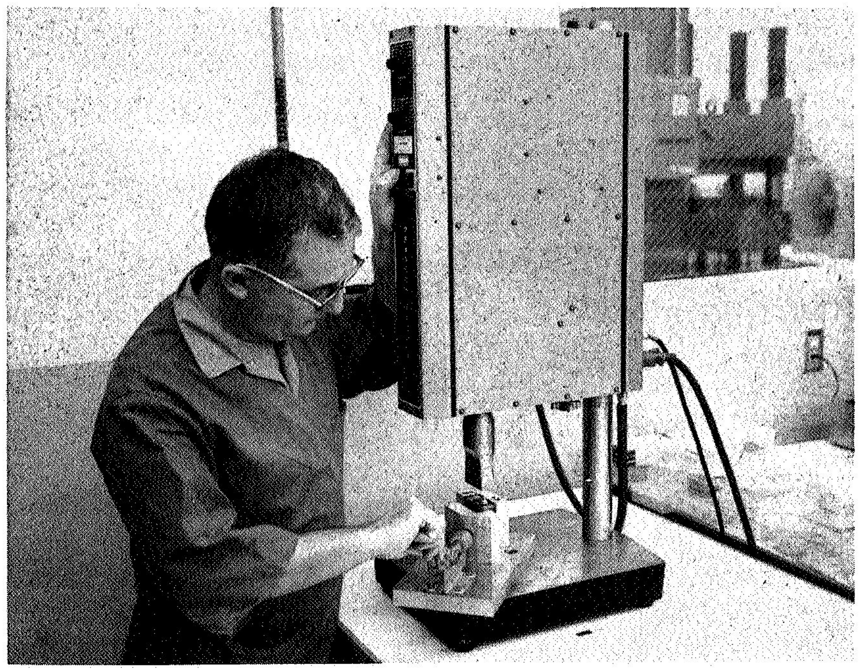

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Figure 9. Ultrasonic Welding of Cover to Case

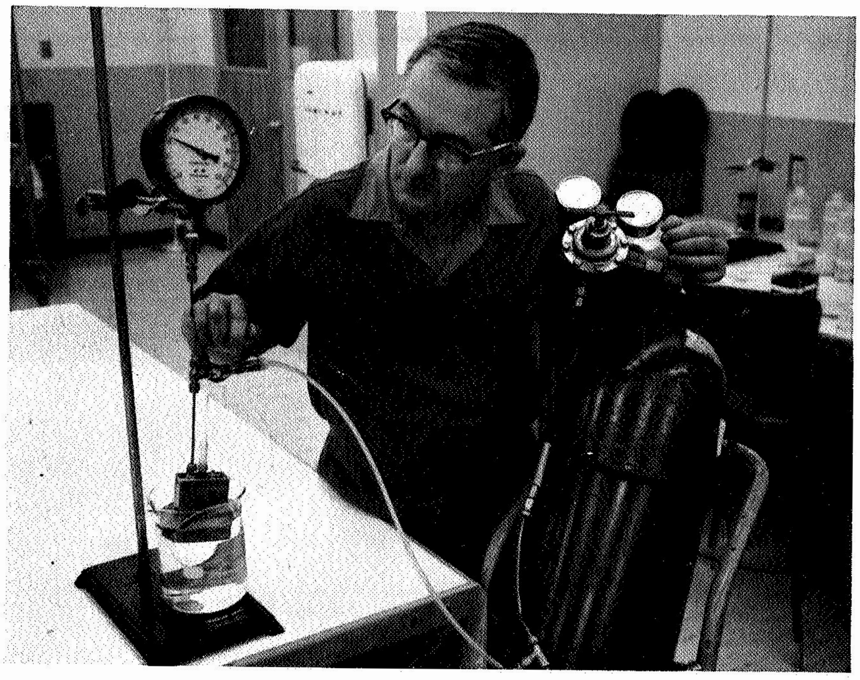

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Figure 10. Pressure Testing of Cell After Welding

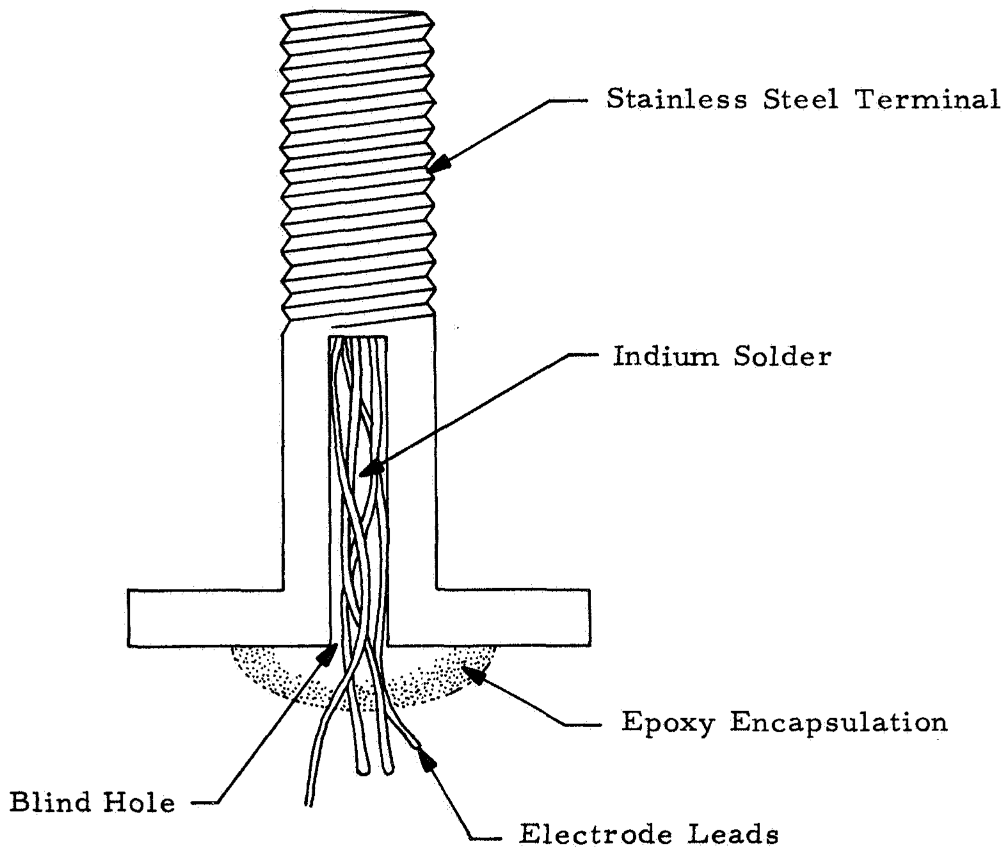

Figure 11. Electrode Lead Attachment to Cell Terminal

As an added protection feature, the base of the terminal is encapsulated in epoxy to cover the exposed solder joint of the leads to the terminal.

3.1.6 Connection Methods and Collectors

Although keeping methods and components similar to those employed in Contract NAS 3-7639 (Reference 1), a small effort was devoted to reinforcing the silver grid to avoid corrosion observed under a continuous cycling at 100°C (Contract NAS 3-7639) which may have been a contributing factor in the cycle life of some cells.

The new grid material selected was a heavier silver exmet, 5 Ag-14-1/0, than the one originally used in the 5-Ah cell, 3 Ag-10-3/0.

3.1.6.1 Corrosion Test

Silver electrodes using the regular and new exmets were assembled in dummy cells, one between two solid nickel sheet counter electrodes, and tested at 100°C under continuous overcharge to determine their ability to stand corrosion.

After 200 hours, silver plates overcharged in free KOH at 100°C against solid nickel counterelectrodes were found extremely warped and corroded, regardless of the silver grid used. The test was repeated with separators and nickel counterelectrodes tightly sandwiching the silver plates as in a normal cell assembly.

It can be pointed out that in the cycling regime of 1/2-hour discharge, 1-hour charge there is no more than nearly 10 minutes of overcharge, which, over 600 cycles, is equivalent to 100 hours of real overcharge. The previous test was therefore considered to be excessively severe. In the repeat test, the progress of corrosion was checked every 48 hours.

It was found that the corrosion and degradation affected only the sintered silver powder, rather than the grid. Weights of grid devoid of silver powder were compared with original weights and were found unchanged for both Exmets.

To prevent the corrosion of the silver leads, ACORN rubber cement was applied around each wire lead filling a Teflon sleeve slipped tightly on the wire down to the base of the wire.

In order to remove all doubts about the effect of a heavier silver exmet grid on the electrical performance at room temperature, the next test was undertaken concurrently.

3.1.6.2 Cell Tests

Five cells were built using the heavier silver exmet, but otherwise of the same design as cells used in Contract NAS 3-7639 (Reference 1).

Three total discharge cycles were given to determine their capacity and voltage uniformity (Table IX). Table X gives their end of discharge voltages at various cycles and a capacity check after 300 cycles. Figures 12 and 13 show their cycling performance at various cycles. They failed at around 2100 cycles because of cracked separators. Upon disassembly, all other components appeared satisfactory. Table XI gives a summary of the cycling data.

3.1.7 Electrolyte

The 30% KOH concentration was used in most tests of Task I. However, a study of the effect of KOH concentration was deemed necessary because it was known from other tests (Reference 3) that the charged wet stand capacity retention is improved with higher electrolyte concentration.

Forty and forty-five percent KOH were used in a group of cells to be compared with the 30% KOH on the same 90-minute period regime. Unfortunately, the cells used had large size silver electrodes (refer to Paragraph 3.1.1.2). Table XII and Figure 14 show the differences caused by the change in electrolyte concentration.

On the 90-minute period regime, the same condition of imbalance prevailed through the automatic cycling (see Paragraph 3.1.1.2) after 432 cycles. The shifting of the positive electrodes was then corrected by immobilization of

TABLE IX

FIVE-AH CELL PERFORMANCE DATA (1 A DISCHARGE)
(HEAVY SILVER EXMET 5 Ag-14-1/0)

Cell No.	Cycle 1		Cycle 2		Cycle 3	
	Output	Voltage	Output	Voltage	Output	Voltage
ZL-1-1	7.5 Ah	1.45 V	7.5 Ah	1.45 V	7.1 Ah	1.45 V
ZL-1-2	7.5	1.46	7.5	1.45	7.0	1.45
ZL-1-3	7.8	1.45	7.2	1.45	6.9	1.45
ZL-1-4	7.7	1.46	7.6	1.45	7.4	1.45
ZL-1-5	7.4	1.45	7.5	1.45	7.1	1.45
Avg.	7.6 Ah	1.45 V	7.45 Ah	1.45 V	7.1 Ah	1.45 V

TABLE X

(Exmet 5 Ag-14-1/0)
VOLTAGE AT END OF DISCHARGE PERIOD (1/2 HOUR)

Cell No.	Cycles				
	5	500	1000	1500	1950
ZL-1-1	1.50	1.46	1.46	1.45	1.45
ZL-1-2	1.50	1.45	1.45	1.44	1.44
ZL-1-3	1.50	1.45	1.44	1.43	—
ZL-1-4	1.50	1.47	1.47	1.46	1.46
ZL-1-5	1.50	1.45	1.45	1.44	1.44
Average	1.50	1.45	1.45	1.44	1.45

300 CYCLE CAPACITY CHECK

Cell No.	Capacity	Average Voltage
ZL-1-1	4.2 Ah	1.34 V
ZL-1-2	3.8	1.33
ZL-1-3	4.0	1.33
ZL-1-4	3.6	1.33
ZL-1-5	5.1	1.33
Average	4.1	1.33

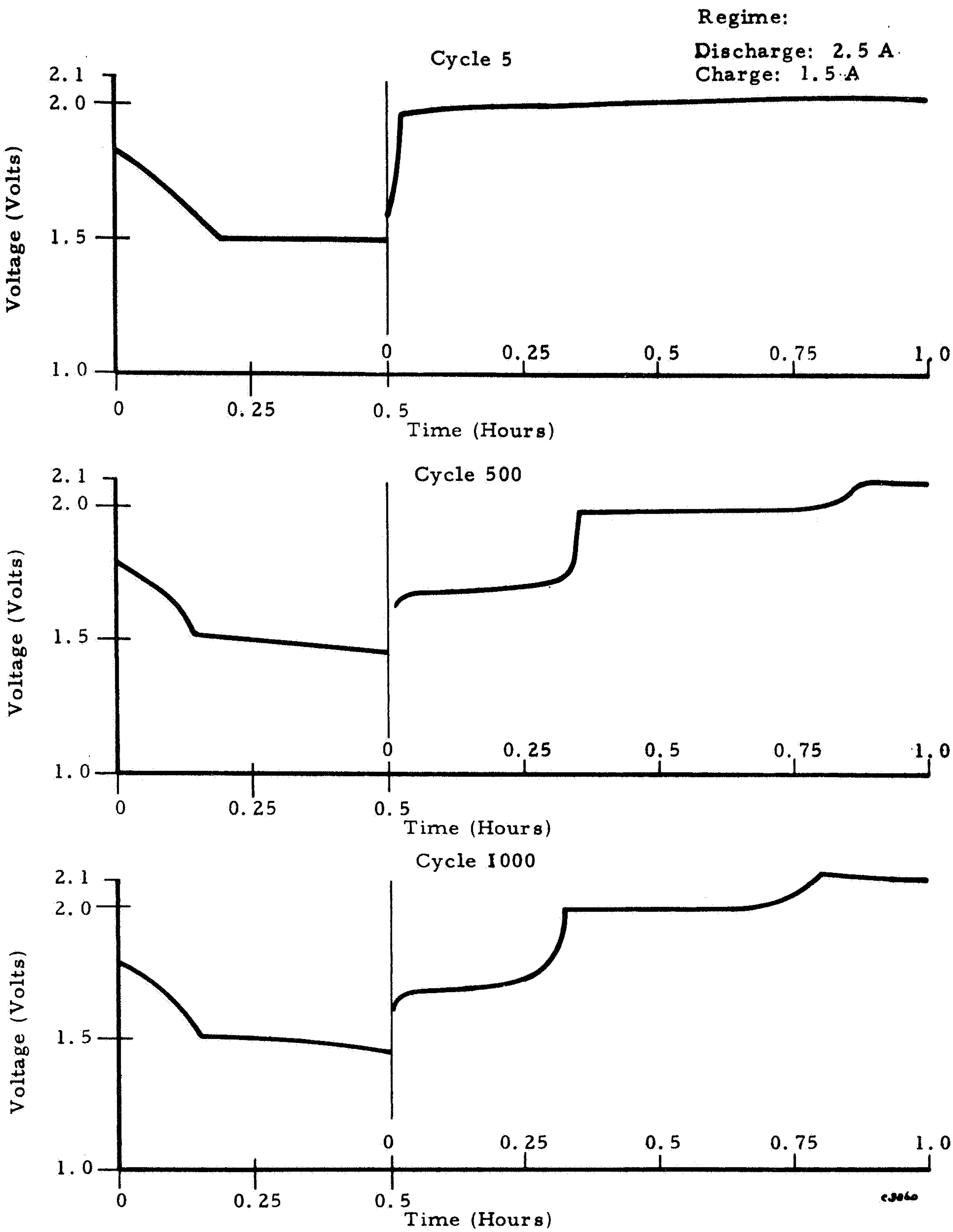

Figure 12. Cycling Curves of Cells With 5 Ag-14-1/0 Exmet

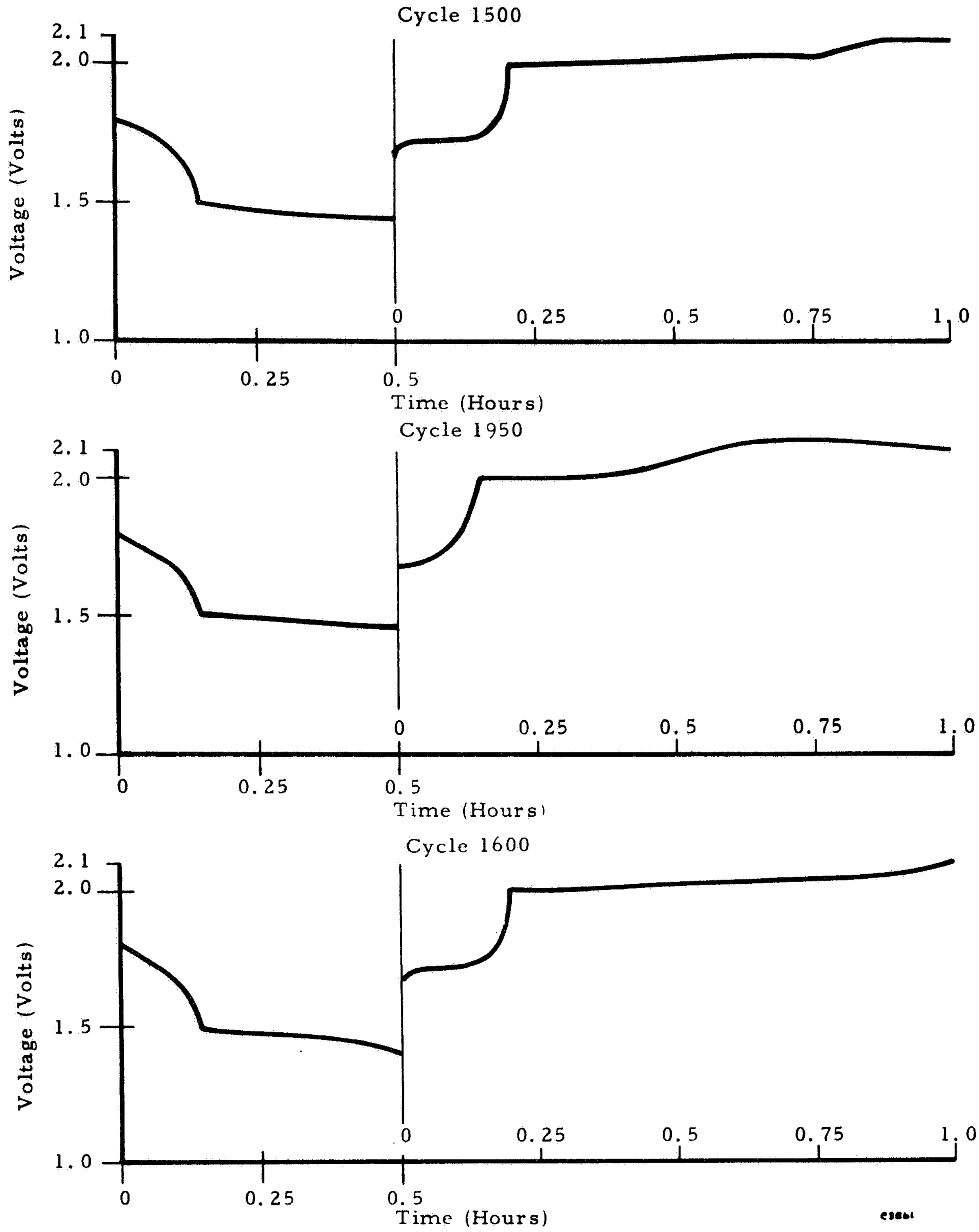

Figure 13. Cycling Curves of Cells With 5 Ag-14-1/0 Exmet

TABLE XI

GROUP ZL-1
CYCLING DATA SUMMARY

Cell No.	Cycles	Final Capacity
ZL-1-1	2300	---
ZL-1-2	2120	1.80 Ah
ZL-1-3	1650	---
ZL-1-4	2147	1.50 Ah
ZL-1-5	2183	---
Average	2080	
Mean Deviation	< 10%	

TABLE XII

FORMATION WITH DIFFERENT KOH CONCENTRATIONS

Design Feature	Cell No.	Output	Plateau Voltage
30% KOH	ZL-25-1	9.66 Ah	1.44 V
	ZL-25-2	9.75 Ah	1.44 V
	Average	9.71 Ah	1.44 V
40% KOH	ZL-25-3	9.41 Ah	1.42 V
	ZL-25-4	9.58 Ah	1.42 V
	Average	9.50 Ah	1.42 V
45% KOH	ZL-25-5	9.00 Ah	1.39 V
	ZL-25-6	8.00 Ah	1.36 V
	Average	8.50 Ah	1.38 V

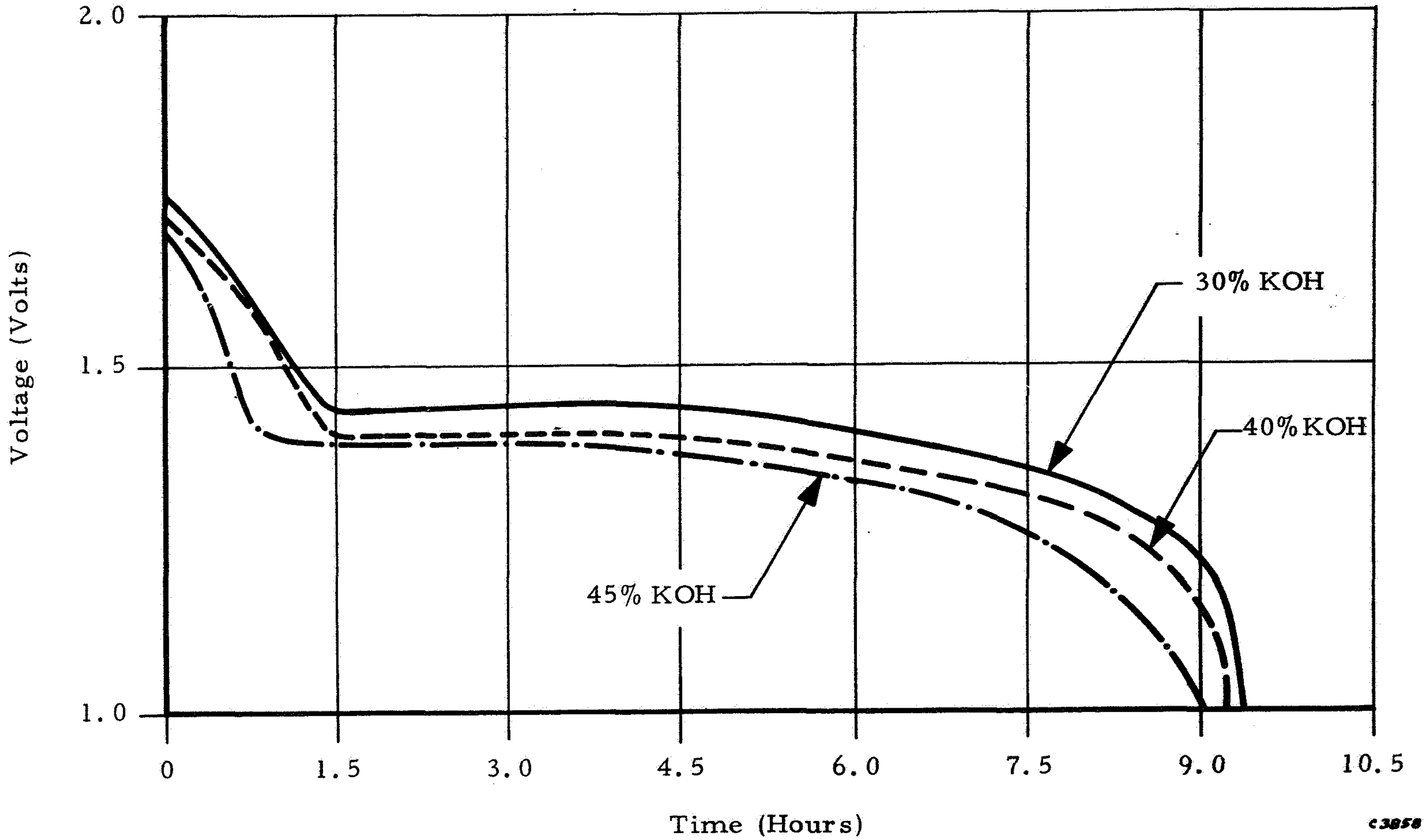

Figure 14. Discharge Curves of Cells With Various Concentrations of KOH

the cell pack with epoxy, thus reverting to the standard positive electrode size.

From the available data, it seems reasonable to use 40% KOH, considering that the capacity and plateau voltage are acceptable and that charged wet stand capacity retention will certainly be improved.

A group of five cells was therefore built integrating all the modifications (cell pack immobilization, 40% KOH electrolyte) and put on test. Table XIII gives their original performance. All data are presented at periodic cycles in the tables entitled "Uniformity Study" giving for each cell of each group:

1. the charge characteristics:

   OC = overcharge percentage

   m% = monoxide plateau percentage of total charge period

   V_f = final voltage at the end of the charge period.

2. the discharge characteristics:

   p% = peroxide plateau percentage of total discharge period

   V_p = plateau voltage

   Ve = end voltage at the end of the discharge period

3. the electrolyte addition in cumulative amounts from the beginning of the test.

Data are reported in Tables XIV, XV, XVI and Figures 15, 16 and 17. When the cells failed to meet the capacity cycling requirement (voltage dropping below 1.0 V before the end of the discharge period), the cells were given a capacity check. Table XVII shows that the capacities were still over 4 Ah, the limit set by the work statement for completion of the test, even after 1300 cycles.

The cells failed the capacity requirement beyond the 1500 cycle mark (Table XVIII). Upon dissection and examination, the separators were not cracked, the electrodes were found completely discharged but in good condition, and the zinc electrodes were not slumped (Figure 18). Some minute shorts may have caused the charge inefficiency. (Note that the cells of this group were not built with the new 3420-09 separator introduced in the last part of Task II.)

TABLE XIII

FORMATION OF STANDARD CELLS WITH 40% KOH ELECTROLYTE

Discharge Rate: 1.0 A to 1.0 V

Cell No.	Capacity	Plateau Voltage
ZL-32-1	7.30 Ah	1.42 V
ZL-32-2	7.80 Ah	1.42
ZL-32-3	7.40 Ah	1.42
ZL-33-4	7.60 Ah	1.42
ZL-34-5	7.80 Ah	1.42
Average	7.60 Ah	1.42 V

TABLE XIV

UNIFORMITY STUDY, GROUP ZL-32

Regime: Discharge: 2.5 A for 1/2 hr
Charge: 1.3 A for 1 hr
Voltage Limit: 2.05 V/cell
Temperature: 25 °C

Cycle 10

Cell Number		1	2	3	4	5	Avg.
Charge	m%	15	15	15	15	15	15
(OC = 5.9 %)	V_f	2.05	2.05	2.05	2.05	2.05	2.05
Discharge	p%	32	32	32	32	32	32
	V_p	1.42	1.42	1.42	1.42	1.41	1.42
	V_e	1.40	1.40	1.40	1.40	1.40	1.40
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

Cycle 300

Charge	m%	29	29	29	29	40	31
(OC = 4.0%)	V_f	2.03	2.04	2.02	2.04	2.00	2.03
Discharge	p%	31	31	31	31	23	30
	V_p	1.31	1.35	1.36	1.35	1.32	1.34
	V_e	1.30	1.30	1.30	1.30	1.29	1.30
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

TABLE XV

UNIFORMITY STUDY, GROUP ZL-32

Regime: Discharge: 2.5 A for 1/2 hr
Charge: 1.3 A for 1 hr
Voltage Limit: 2.05 V/cell
Temperature: 25°C

Cycle 600

Cell Number		1	2	3	4	5	Avg.
Charge	m%	19	19	19	19	19	19
(OC = 9.6 %)	V_f	2.08	2.04	2.03	2.05	2.00	2.04
Discharge	p%	30	30	30	30	30	30
	V_p	1.30	1.32	1.33	1.31	1.33	1.32
	V_e	1.27	1.28	1.31	1.29	1.30	1.29
Electrolyte Addition	Cum. Amt (cc)	2.0	2.0	3.0	3.0	3.0	2.6

Cycle 900

Charge	m%	18	18	18	18	18	18
(OC = 9.0 %)	V_f	2.07	2.06	2.08	2.07	2.07	2.07
Discharge	p%	29	29	29	29	29	29
	V_p	1.30	1.33	1.33	1.31	1.34	1.32
	V_e	1.25	1.25	1.26	1.22	1.27	1.25
Electrolyte Addition	Cum. Amt (cc)	5.0	7.0	5.0	4.5	3.0	4.9

TABLE XVI

UNIFORMITY STUDY, GROUP ZL-32

Regime: Discharge: 2.5 A for 0.5 hr
Charge: 1.3 A for 1.0 hr
Voltage Limit: 2.05 V/cell
Temperature: 25°C

Cycle 1200

Cell Number		1	2	3	4	5	Avg.
Charge	m%	18	18	29	18	20	21
(OC = 8.3%)	V_f	2.03	2.09	1.98	2.06	2.01	2.03
Discharge	p%	34	37	26	34	37	34
	V_p	1.30	1.35	1.34	1.32	1.33	1.33
	V_e	1.28	1.32	1.31	1.30	1.32	1.31
Electrolyte Addition	Cum. Amt (cc)	7.5	10.5	9.5	10.0	5.5	8.6

Cycle 1500*

Charge	m%	14	17	18	17	20	17
(OC = 5.2%)	V_f	2.12	2.12	2.14	2.04	2.16	2.12
Discharge	p%	35	34	35	33	30	33
	V_p	1.26	1.24	1.30	1.24	1.30	1.27
	V_e	1.16	1.16	1.20	1.20	1.24	1.19
Electrolyte Addition	Cum. Amt (cc)	13.0	17.5	19.5	16.5	12.0	15.7

*At cycle 1330, current and voltage limits were increased to 1.5 A and 2.12 V/cell, respectively.

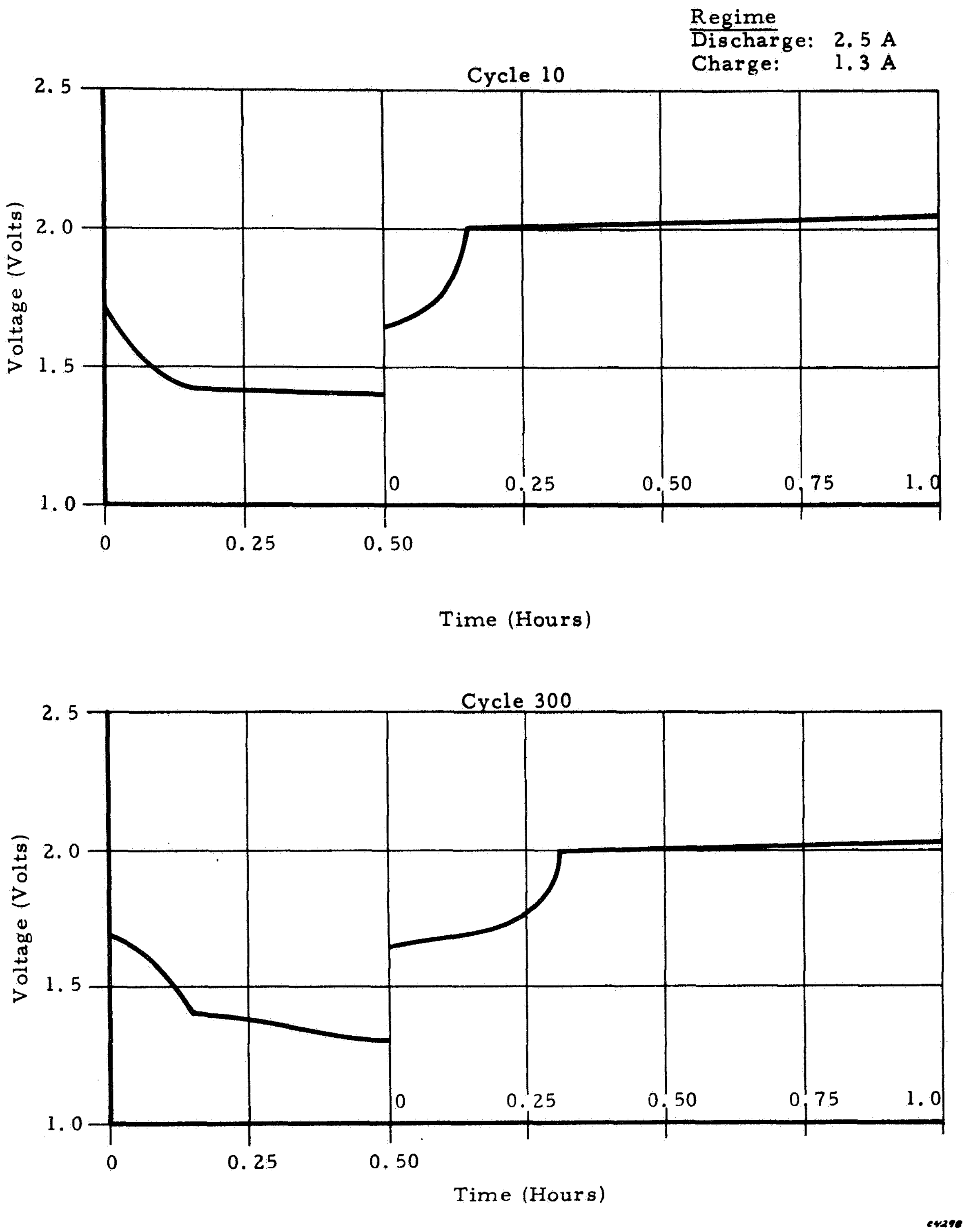

Figure 15. Group ZL-32 Typical Cycling Curves

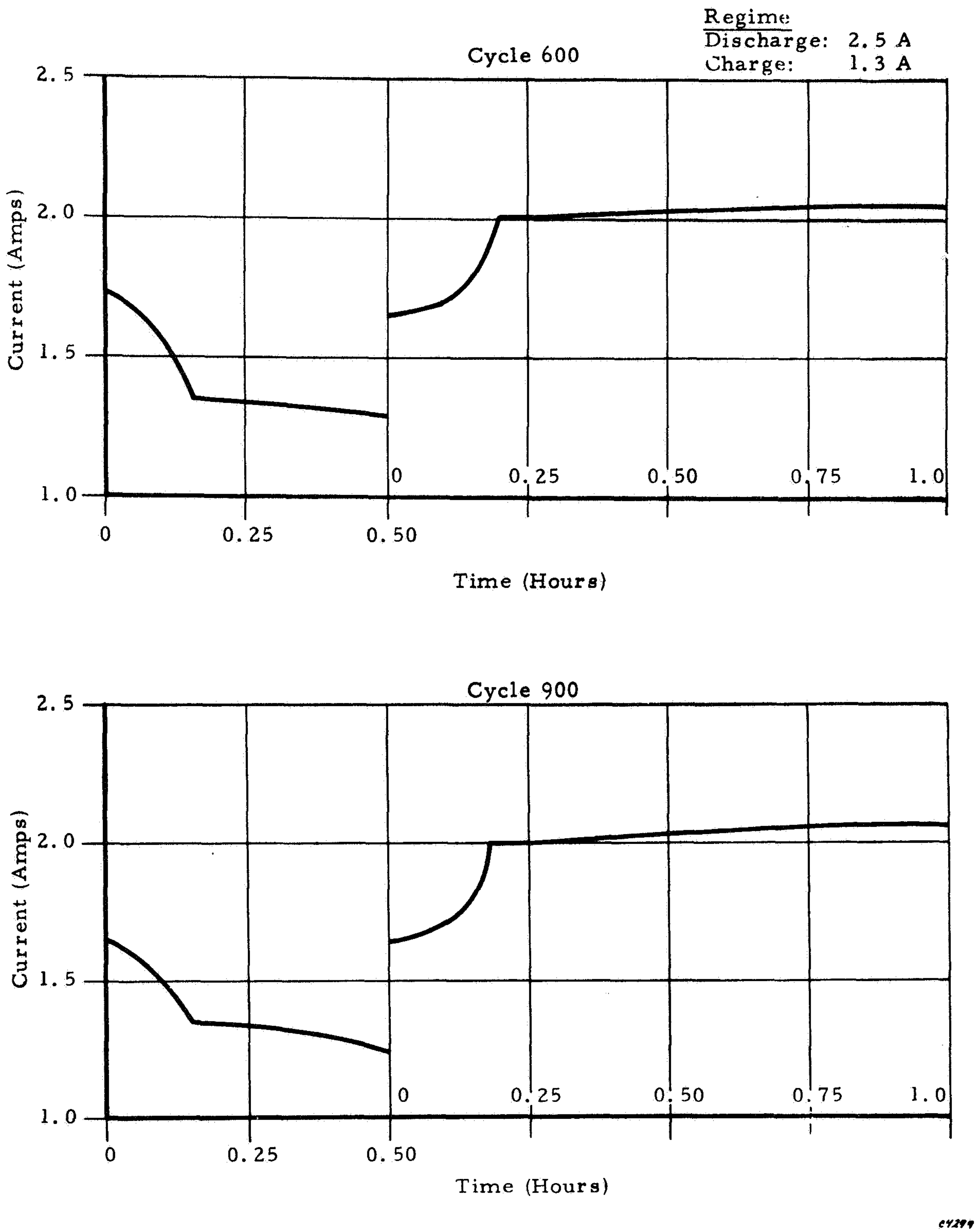

Figure 16. Group ZL-32 Typical Cycling Curves

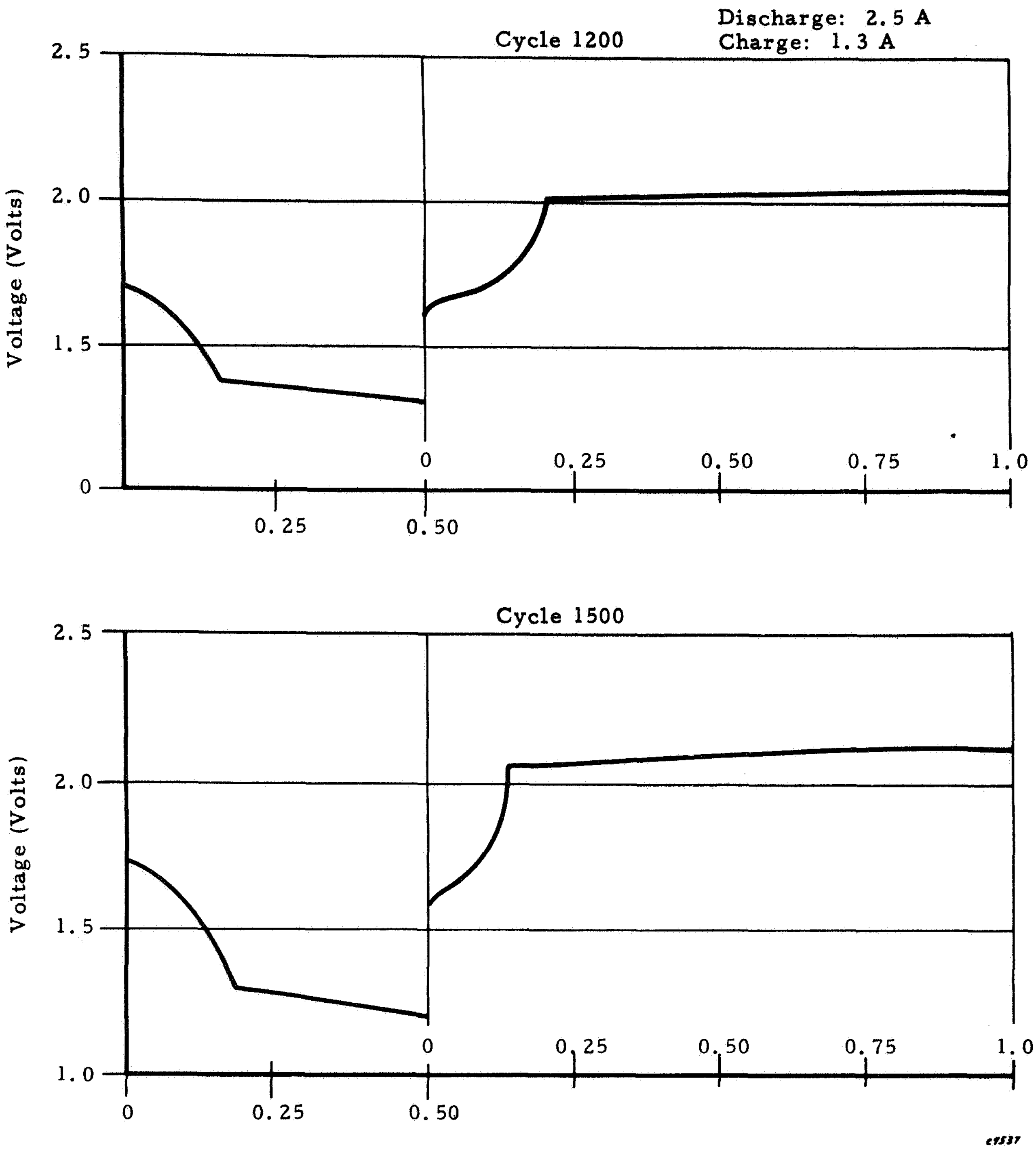

Figure 17. Group ZL-32 Typical Cycling Curves

TABLE XVII

GROUP ZL-32 CAPACITY CHECK DURING THE 90-MINUTE CYCLING

Cycle No.	Capacity				
	Cell #1	Cell #2	Cell #3	Cell #4	Cell #5
1152		4.0 Ah			
1160			4.5 Ah		5.0 Ah
1174	5.0 Ah			4.9 Ah	
1237	4.25 Ah				
1258					4.7 Ah
1326	4.50 Ah				
1336		4.15 Ah			
1341				4.8 Ah	
1342			4.6 Ah		
1377			5.3 Ah		
1492				4.7 Ah	
1504			3.75 Ah		
1571	2.7 Ah				
1583				2.5 Ah	
1628		1.6 Ah			
1632					3.8 Ah

TABLE XVIII

GROUP ZL-32 - TEST SUMMARY

Cell No.	Original Capacity (Ah)	Total Cycles	Final Capacity (Ah)
1	7.30	1571	2.70
2	7.80	1628	1.60
3	7.40	1504	3.75
4	7.60	1583	2.50
5	7.80	1632	3.75
Average	7.60	1583	2.80
Deviation	<3%	<4%	35%

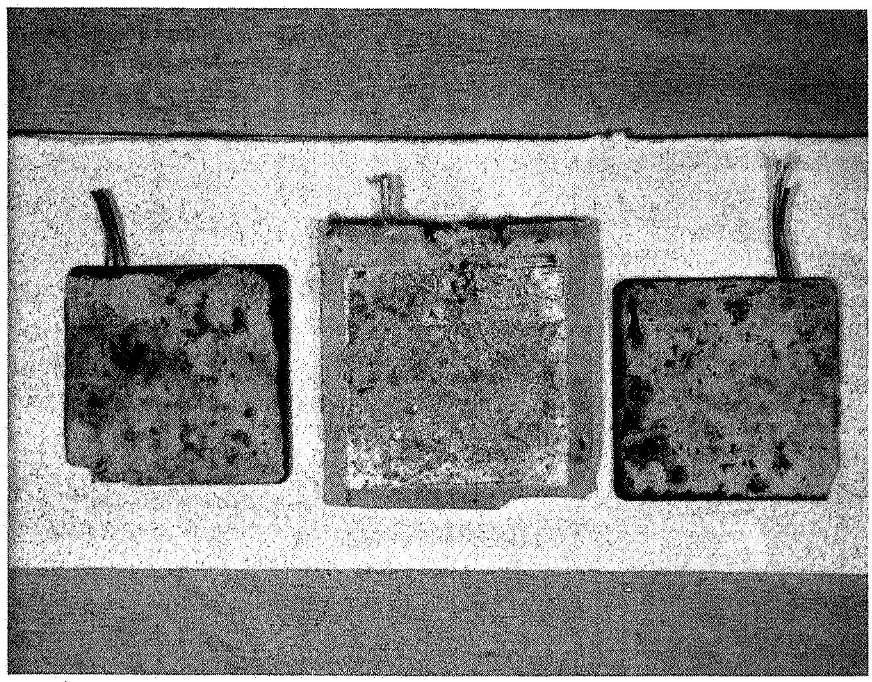

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Figure 18. ZL-32 Cell Electrode Set After 1632 Cycles. Bottom corners were chipped during dis-assembly.

TABLE XIX

MATERIAL TESTS

(Average of 2 samples tested for 200 hours at indicated temperatures)

Item	Material (sin, Hardener Ratio)	Test Condition	Color	Surface	Hardness: Immediately After Test	Hardness: After Washing and Drying	% Weight Change
1	Allbond w/filler (control)	Original	Grey	Smooth	—	—	0
		25°C	NC	Smooth	NC	NC	-0.18
	50/50	100°C	sl. darker	sl. pitted	NC	NC	-0.23
		135°C	sl. darker	sl. pitted	NC	NC	-0.22
2	Allbond unfilled	Original	amber	smooth	—	—	—
		25°C	light brown	smooth	NC	NC	-0.11
	64/86	100°C	dark brown	sl. pitted	NC	NC	+0.07
		135°C	dark brown	sl. pitted	NC	NC	+0.43
3	Epon 815	Original	light yellow	smooth	—	—	—
		25°C	light yellow	smooth	NC	NC	-0.27
	94/6 A	100°C	dark yellow	smooth	NC	NC	+0.32
		135°C	dark yellow	rough	NC	NC	+0.37
4	Epon 815	Original	light yellow	smooth	—	—	—
		25°C	light yellow	smooth	NC	NC	-0.02
	83/17 T-1	100°C	dark yellow	gummy	soft	NC	+0.01
		135°C	dark yellow	gummy	very soft	NC	+0.47
5	Epon 815	Original	light yellow	smooth	—	—	—
		25°C	light yellow	smooth	NC	NC	-0.00
	77/23 T-1	100°C	dark yellow	gummy	soft	NC	-2.06
		135°C	dark yellow	degraded loose material	very soft	NC	-4.19

NC = No Change
sl. = slightly

(Continued)

TABLE XIX (Concluded)

MATERIAL TESTS

(Average of 2 samples tested for 200 hours at indicated temperatures)

Item	Material (Resin, Hardener Ratio)	Test Condition	Color	Surface	Hardness: Immediately After Test	Hardness: After Washing and Drying	% Weight Change
6	Epon 815 92/8 A	Original	light yellow	smooth	—	—	—
		25°C	light yellow	smooth	NC	NC	-0.5
		100°C	yellow	smooth	NC	NC	+0.2
		135°C	dark yellow	sl. pitted	NC	NC	-2.4
7	Epon 815 90/10 A	Original	light yellow	smooth	—	—	—
		25°C	light yellow	smooth	NC	NC	-0.4
		100°C	yellow	smooth	NC	NC	-0.5
		135°C	dark yellow	rough	NC	NC	-5.8
8	Acorn #724 Rubber Cement	Original	orange	smooth, some bubbles	—	—	—
		25°C	dark red	smooth, some bubbles	NC	NC	+0.9
		100°C	light yellow	smooth, some bubbles	NC	NC	-1.9
		135°C	dark yellow	smooth, some bubbles	NC	NC	-4.8
9	Chloroprene Rubber	Original	black	smooth	—	—	—
		25°C	black	sl. pitted	sl. harder	NC	-7.1
		100°C	black	sl. pitted	sl. harder	NC	-9.9
		135°C	black	smooth	sl. harder	NC	-11.2
10	Silastic Rubber 35 (Dow Chemical)	Original	light tan	smooth	—	—	—
		25°C	light tan	smooth	NC	NC	-0.3
		100°C	light tan	smooth	NC	NC	-6.7
		135°C	tan	rough dissolved	NC	NC	-81.2
11	Silastic Rubber 55 (Dow Chemical)	Original	tan	smooth	—	—	—
		25°C	tan	smooth	NC	NC	-0.3
		100°C	tan	smooth	NC	NC	-12.7
		135°C	tan	dissolved			

3.1.8 Separator Edge Seals

To select a material for the separator edge seal that may be capable of resisting chemical attack by KOH at elevated temperatures (as high as 135°C) for 200 hours, several formulations were considered. Material tests and bond tests were made.

3.1.8.1 Material Tests

Samples of each material (2" in diameter by 0.5" thickness) were made and, after measuring their physical characteristics (dimensions, volume, and weight), were submitted to 45% KOH for 200 hours at 25°, 100°, and 135°C.

The list of materials tested and the test results are reported in Table XIX.

The epoxy type Allbond originally used (control) and Epon 815 with hardener not higher than 8% appeared promising. Of the elastomeric types, ACORN #724 rubber cement is the only one that may offer a possibility (see Paragraph 3.1.8.3).

3.1.8.2 Bond Tests

To determine the figure of merit of separator edge seals under dry and wet (KOH) conditions at temperatures as high as 135°C, attempts were made to measure the peel strength and shear strength of the epoxy-bonded separator over a bonded area of the same width as the edge seal area of the wafer construction (i.e., 1/4").

1. For comparative and correlative purposes, the 3420-09 inorganic separator was tested by itself to determine its modulus of rupture and its tensile strength. The results are shown in Table XX.
2. For the peel strength test, no satisfactory method was found suitable because the separator breaks transversally before the epoxy bond peels off.
3. For the shear strength test, a special shearing jig was set up as depicted in Figure 19. Each separator was cemented to a metal backing plate to avoid breaking the separator under tensile pull. Two samples of Allbond epoxy-bonded separators (control) were tried dry at 25°C. The separators broke under tensile pull, sliding away from the metal plates. The entire bonded separator areas remained intact. The values obtained (about 6000 psi) represent the tensile strength of the epoxy-filled separator.

TABLE XX

SEPATATOR 3420-09 PHYSICAL CHARACTERISTICS

	Modulus of Rupture* (psi)	Tensile Strength (psi)
	9150	2040
	9190	1850
	9180	2020
	9100	
	8990	
	9100	
	9170	
	9000	
	8500	
Average:	9040	1970
Mean Deviation:	±135	±80

*See Appendix C for description of the experimental determination.

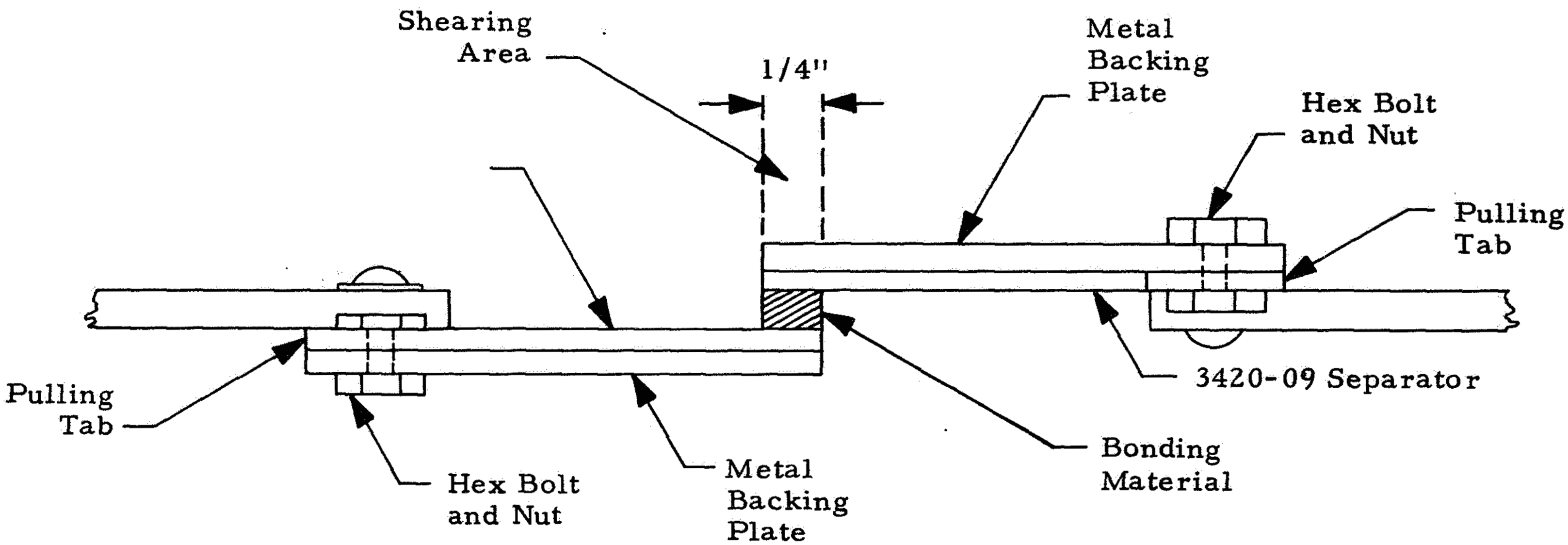

Figure 19. First Concept for Epoxy Separator Bond Shear Test

A new set-up was devised to determine the true value of the shear strength of the bond (Figure 20). The tests were run on bonded separator samples in the dry condition and in the wet condition (immersion in 45% KOH for 200 hours) at 25°C and 100°C (Table XXI). Generally, the oven cure and the separator edge impregnation before bonding improve the adhesiveness. On the basis of these data, the Epon 815 with 8% hardener A appeared to be a little superior to the control Allbond up to 100°C. However, the wide scatter of data does not support a firm conclusion.

Tests at 135°C were run on selected compositions only in conditions duplicating actual cell assembly where the bonded areas are under compression. Table XXII shows a better average for Epon, but here again the range of data for both epoxies is quite wide.

Because the cell is not intended for operation over 100°C, and based on the extensive experimental evidence of reliable usage in actual cells at temperatures up to 100°C, Allbond epoxy as used previously is therefore retained as the separator edge sealant.

3.1.8.3 Cells with Separator Edges Sealed with Rubber Cement

Recognizing that the bond tests were not too meaningful with an elastomeric seal, six cells were made for actual test purposes. They were built with chloroprene rubber gasket cemented with Acorn 724 to seal the separator edges.

For fast evaluation, five cells were tested at 100°C on the 1/2-hour, 1-hour cycling regime. After a few cycles, electrolyte spewage occurred, causing premature drying. After refilling, the cells kept cycling but with constant electrolyte spewage and foaming. The cells were stopped after 80 cycles for dissection and examination. The separators were not cracked, but delamination of the seals was common to all wafers. Greenish foam noted in the electrolyte and pinkish color on the positives led to the belief that the chloroprene was electrochemically attacked.

Although the cell was not designed for heat sterilization because of several features incorporated in the design (Pellon interseparator, inorganic separator

TABLE XXI

SHEAR STRENGTH OF BONDED SEPARATORS

Item	Material (resin, hardener)	Dry reading	Dry average	After exposure to KOH for 200 hours at 25°C reading	at 25°C average	at 100°C reading	at 100°C average
1	Allbond with	2690					
	filler (edge	4150					
	impregnated)	2400					
	50/50	4100					
		2690					
		3090					
		4490					
		2240					
		3890					
		3060		370		420	
		4090		510		390	
		3300		410		400	
		2040		310		830	
		2210		800		125	
		2440	3100	410	470	125	380
1A	Allbond with	2600					
	filler (edge	3090	2770				
	not impregnated)	2610					
	50/50						
2	Allbond unfilled	3480		1310			
	(edge	3430		1470			
	impregnated)	3850		540		250	
	64/36	3560		560		460	
		3620	3790	320	840	310	340
2A	Allbond unfilled	2830					
	(edge not	2980					
	impregnated)	2240					
	64/36	2240					
		2400	2540				
3	Epon 815	2240					
	94/6A	2240					
	R. T. cure	2240					
		2560	2320				
	Epon 815	2640					
	94/6A	2650		650			
	oven cure	2690		790			
		2650		750		470	
		2690		370		190	
		2690		490		180	
		2850	2700	620	610	310	290

(Continued)

TABLE XXI (Concluded)

SHEAR STRENGTH OF BONDED SEPARATORS

Item	Material (resin, hardener)	Dry reading	Dry average	After exposure to KOH for 200 hours at 25°C reading	at 25°C average	at 100°C reading	at 100°C average
6	Epon 815	2560					
	94/8A	2290					
	R. T. cure	3070					
		1810	2430				
	Epon 815	2700		1270			
	94/8A	2850		310		210	
	oven cure	3130		670		610	
	(edge not	3600		1780		110	
	impregnated)	4350		1100		1230	
		4390	3500	620	790	440	520
6A	Epon 815	2560		790		260	
	94/8A	2860		840		420	
	oven cure	2800		440		1380	
	(edge	2830		1000		230	
	impregnated)	2180		1330		200	
		3740	2830	980	900	640	520
7	Epon 815	1920					
	90/10A	1230					
	R. T. cure	2770					
		1890					
		1230					
		1390	1740				
	Epon 815	3360					
	90/10A	2270		760			
	oven cure	2680		580		440	
		4220		640		460	
		3630		550		190	
		3040		750		540	
		3630	2860	580	640	330	390
8 and 9	Chloroprene	470		-		-	
	rubber gasket	530		-		-	
	cemented with	150		-		-	
	ACORN 724	600		-		-	
		140		-		-	
		180	350	25*		25*	

* estimated

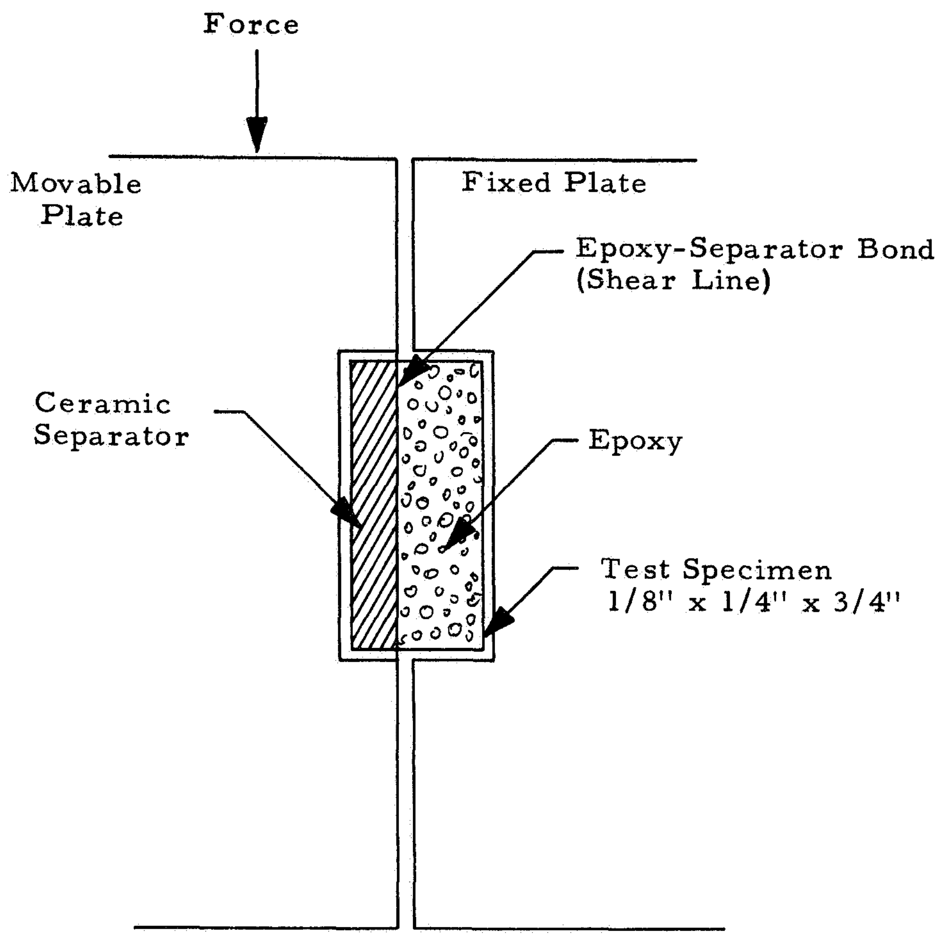

Figure 20. Second Concept for Epoxy-Separator Bond Shear Test

TABLE XXII

SHEAR STRENGTH OF BONDED SEPARATORS SUBMITTED TO KOH AT 135°C FOR INDICATED HOURS (psi)

Item	Material	120 Hours			170 Hours			
		Readings	Range	Average	Readings	Range	Average	% over 130 psi
1	Allbond with filler (edge impregnated 50/50	150 105		130	76 15 15 457 428 18	15-457	170	33%
6 A	Epon 815 94/8A oven cure (edge impregnated)	555 210		380	204 148 506 41 477 161	41-506	260	83%

not optimized for sterilization, cell not hermetically sealed), the remaining cell built with elastomeric edge sealant was heat sterilized at 135°C for 200 hours after being fitted with a 40 psig pressure relief valve and placed in a sealed chamber. Upon termination of the test, moisture was noted condensed on the internal walls of the chamber and was found to be neutral when tested with indicator paper.

The cell was then charged at 350 mA and was carried to 2.3 V cutoff voltage to get an input of 7 Ah. On discharge, the output at 1 A was 4.25 Ah with a plateau voltage of 1.18 V. During recharge, electrolyte spewage occurred with the green foam characteristics noted in the other five cells.

The cell was disassembled and the components examined.

1. The valve was checked: the first cracking pressure was 53 psi and the reseating 29 psi. The next four consecutive cracking pressures were 41 psi and reseating pressures 30 psi. The valve was disassembled. The rubber disk was intact and in good condition.
2. The wafers were in good condition — no delamination of the seals or collars, no cracks in the separators.
3. The electrodes were in good condition. The KT interseparator was intact, but the Pellon interseparator (nylon felt) was completely deteriorated.

In an overall evaluation, most of the components appear suitable for heat sterilization. As expected, the Pellon nylon felt is unsuitable, but can be replaced if need be. The separator edge seals appear capable of heat sterilization, but not suitable for cycling conditions. Although functional, the separator exhibited an increase in electrical resistance, translated by the decline in plateau voltage and capacity from the original unsterilized state. This was expected, as this separator 3420-09 was never recommended for heat sterilizable cells, because it lacks the requirement of low resistivity.

Since the present program is not concerned with heat sterilized cell development, further work along this line was discontinued as ordered by the NASA Program Manager (Reference 4).

3.1.9 Valves

In order to approximate the ambient conditions in actual cells, the valve evaluation was done in a KOH saturated atmosphere, with pressure up to 40 psig and temperatures up to 100°C. The testing procedure was set as follows:

1. The valve is adapted to the cover of a sealed and constrained plastic case capable of pressurization up to 50 psig. KOH is introduced into the empty case to approximately the same level as in a regular 5-Ah cell. The set-up has a pressure gauge and a fitting for introducing nitrogen under pressure.
2. The internal pressure is built up with nitrogen to cause the valve to open (the cracking pressure is noted) and to reclose upon loss of pressure (the reseating pressure is noted).
3. Ten consecutive observations are made 15 minutes apart every day over a period of 30 days.

Three different conditions were used:

25°C — upright position

25°C — occasional changes in attitude to cause KOH to come in contact with the valve

100°C — upright position

A few valves set at 40 psig cracking pressure were obtained from two suppliers, A and B, and our own shop (Douglas type C). Three valves of each model were tested. Because the valves are relatively expensive and are not considered an important factor in this program, the NASA Project Manager recommended another inexpensive valve, AA, to be tested. Average test results are presented in Tables XXIII, XXIV, XXV, and XXVI, respectively, for types A, B, C, and AA.

The valves appeared acceptable for room temperature operation on this program. The relatively inexpensive type AA were ordered for all cells required for the remainder of the program.

As this valve is a little too large for properly fitting in the present cover, being extremely close to the terminals, a new cover mold was ordered with terminal holes further apart so as to avoid the possibility of a short through the valve. At the same time, the new molded cover was provided with a threaded center hole to accept the selected valve body.

TABLE XXIII

AVERAGE TEST DATA OVER 30 DAYS

VALVE A

Test Conditions		Cracking Pressure	Reseating Pressure
Temperature	Attitude		
25°C	Upright	39 psig	37 psig
25°C	Occasional Changes	failed after	16 days
100°C	Upright	36 psig	29 psig

TABLE XXIV

AVERAGE TEST DATA OVER 30 DAYS

VALVE B

Test Conditions		Cracking Pressure	Reseating Pressure
Temperature	Attitude		
25°C	Upright	43 psig	33 psig
25°C	Occasional Changes	42 psig	31 psig
100°C	Upright	37 psig	31 psig

TABLE XXV

AVERAGE DATA FOR VALVE C

Test Conditions		Cracking Pressure (psig)	Reseating Pressure (psig)	Notes
Temperature	Attitude			
25°C	Upright	43	36	1
25°C	Occasional Changes	49	37	2
100°C	Upright	50	25	3

Notes:

1. Valve completed 30-day test.
2. Last reading before valve failed to crack after 9 days.
3. Last reading before valve failed to reseat after 9 days.

TABLE XXVI

AVERAGE TEST DATA OVER 30 DAYS

VALVE AA (INEXPENSIVE MODEL)

Test Conditions		Over 30-Day Test Period		After 30-Day Test Period	
Temperature	Attitude	Cracking Pressure (psig)	Reseating Pressure (psig)	Cracking Pressure (psig)	Reseating Pressure (psig)
25°C	Upright	41	36	41	36
25°C	Occasional Changes	39	0[a]	39	0[a]
100°C	Upright	40	41	41	27

[a]Valve would not seat until pressure dropped to zero psig.

3.1.10 Gassing Study

The objective of this study was to determine the gassing characteristics of the cells under various cycling conditions:

Group I: 25°C, 2-hour period regime
Group II: 25°C, 24-hour period regime
Group III: 40°C, 2-hour period regime

Each group consisted of 10 cells:

1. Five cells in a free-venting set up: gas was collected under inverted graduated cylinders.
2. Five cells in a pressurized condition: the cells were fitted with 40 psig relief valves and pressure gauges, with provision for gas venting and collection past the valve. One cell of this group was connected to a pressure transducer to record the pressure variations continuously.

All cells had provision for sampling the collected gas to determine its composition. Figure 21 shows the set-up for one group.

3.1.10.1 Group I (25°C, 2-hour period)

The first 10 cell-group was tested at 25°C on the 2-hour period regime: 3 A discharge for 35 minutes, 1.4 A charge for 85 minutes.

The average gassing rate increased rapidly in the first 80 cycles to approximately 20 cc/hr and remained near this value for the remainder of the test (Tables XXVII and XXVIII). Average gassing rates and average cumulative gassing of pressurized cells were substantially lower than that of the free venting cells. The gassing rate also increased when the cell voltage limit was increased from 2.03 V to 2.10 V on Cycle No. 105 (Figure 22). This increase in the voltage limit of the charge cycle was necessary to replenish the energy removed on discharge. The gas collected during this test was almost entirely hydrogen.

After 207 cycles, the cells were all opened and the electrolyte readjusted. An average of 8 cc of electrolyte, equivalent to 9 liters of hydrogen gas, was required per cell. Average cumulative cell gassing, including that from formation and open circuit stand amounted to close to 9 liters within 5%.

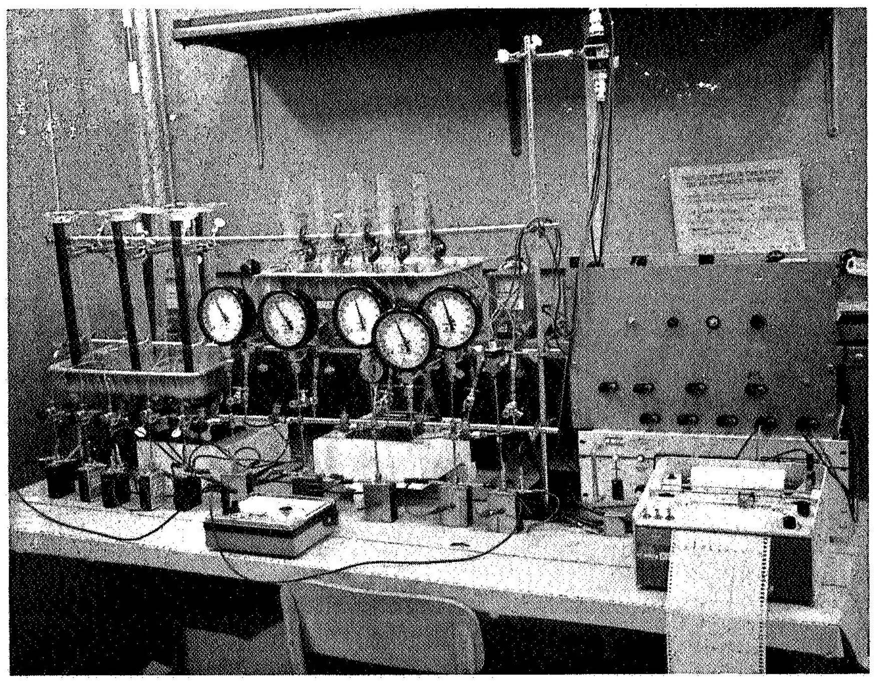

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Figure 21. Gassing Test Set-Up

TABLE XXVII

GASSING TESTS

Group I: Temperature: 25°C
3 A discharge for 35 minutes
1.4 A charge for 83 minutes
Average voltage limit 2.1 V/cell

AVERAGE DATA AS A FUNCTION OF CYCLES

Time Elapsed (hrs)	Cycle No.	Free-Venting Cells: Gas Composition N_2 (%)	O_2 (%)	H_2 (%)	Gassing Rate (cc/hr)	Cumulative Gas Volume (cc)	Pressurized Cells: Gas Composition N_2 (%)	O_2 (%)	H_2 (%)	Pressure at end of Charge (psig)	Cumulative Vented Gas (cc)
0	0	23	57	20	—		14	76	10	—	—
6	3	25	44	31	0.0	0.0	14	72	14	0.0	0.0
24	12	15	25	60	0.6	5.8	12	47	41	1.5	0.0
46	23	8	6	86	0.8	25.0	15	85*	0	—	—
52	26	8	6	86	1.8	38.7	19	20	11	10.0	0.0
72	36	3	1	96	1.9	73.0	9	10	81	15.4	0.0
96	48	1	4	95	3.8	153.0	7	11	82	32.5	35.6
140	70	1	—	99	6.5	521.0	3	14	83	30.0	274.0
164	82	—	—	100	21.3	900.0	2	3	95	33.0	453.0
204	102	—	—	—	16.9	1702.0	—	—	—	33.8	760.0
392	196	—	—	—	20.5	5705.0	—	—	—	33.2	4076.0
460	230	1	8	91	22.3	7275.0	3	8	89	40.8	5623.0
520	260	—	—	—	20.0	8733.0	—	—	—	40.0	7603.0
600	300	—	—	—	20.5	10,123.0	—	—	—	40.0	8866.0
660	330	—	—	—	19.9	11,388.0	—	—	—	40.0	9769.0
720	360	—	—	—	13.4	12,546.0	—	—	—	40.0	11,225.0

*Pressurized cells were purged with oxygen.

NOTES:
1. All cells were purged with oxygen at cycle 207 when electrolyte was added.
2. Average charging voltage limit per cell was changed from 2.03 V to 2.10 V at cycle 101 to permit efficient charge to discharge ratio for continuous cycling.

TABLE XXVIII

GASSING TESTS

Group I = Temperature = 25°C
3 A discharge for 35 minutes
1.4 A charge for 85 minutes
Voltage limit 2.03 V/Cell

STATUS OF INDIVIDUAL CELLS AT CYCLE 70

Test	Cell No.	Accumulated Gas Volume (cc)	Gas Composition N_2 (%)	Gas Composition O_2 (%)	Gas Composition H_2 (%)	Pressure at end of Charge (psig)
Free-Venting	ZL-22-1	270	—	—	100	—
	ZL-22-2	655	—	—	100	—
	ZL-22-3	490	4	1	95	—
	ZL-22-4	610	—	—	100	—
	ZL-22-5	700	—	—	100	—
Pressurized	ZL-22-6	263	4	20	76	30
	ZL-22-7	459	5	4	91	30
	ZL-22-8	111	2	7	91	20
	ZL-22-9	275	2	16	82	35
	ZL-22-10	264	2	21	77	35

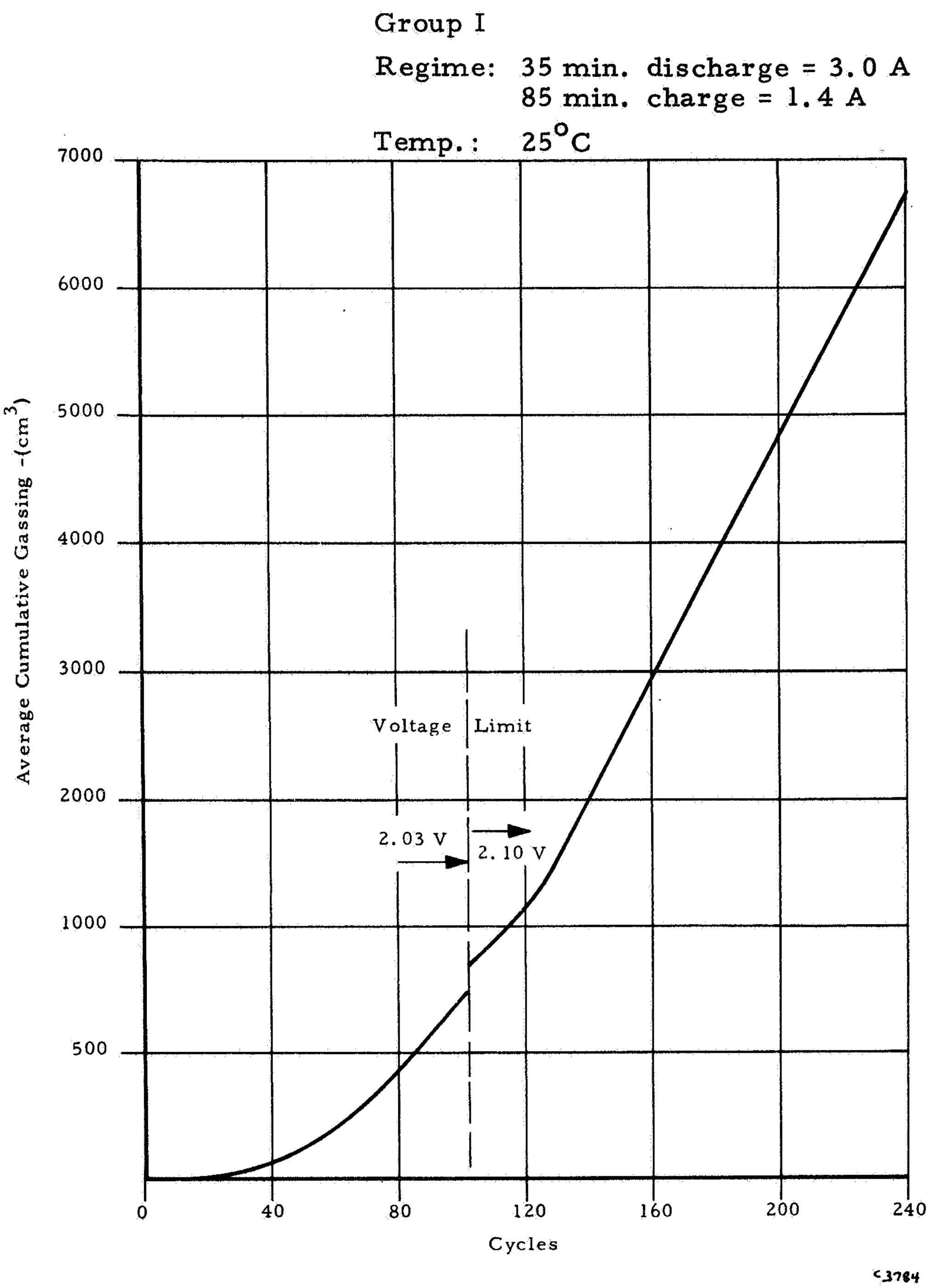


Figure 22. Gassing vs. Cycle (5 Ah Ag-Zn Cells)

This first group of 10 cells completed 360 cycles as scheduled on the 2-hour cycling regime. The data are presented in Table XXVII. Figure 23 shows the variation of the average gassing rate on cycling on this regime. Figure 24 shows the variation of the average gassing rate on charged stand at various times during the cycle life of the cells. Table XXIX gives a comparison of the amount of gas collected and measured versus the gas equivalent of electrolyte consumed during the entire testing period (including gas generated during stand). The total wet life of these cells was 80 days from formation to the last capacity checks. Barring possible leaks during the cycling, assembly and disassembly of the experimental set up, it appears that the gas collected was in all cases smaller than the gas equivalent of the water consumed. This coupled with the fact that the major percentage of gas composition was hydrogen in practically the entire life of the cells shows that some oxygen must have recombined with the zinc, more so in pressurized cells than in free-venting cells.

The cells after completing their cycling were checked for capacity and OCV. Immediately after 360 cycles, they were removed from cycling at the end of charge, then discharged to 1.0 V at the cycling rate 3 A, then drained to 1.0 V at 0.5 A for determining the total capacity.

The cell capacity was low to medium with a good OCV. A normal charge to 2.05V cut-off voltage could not restore all the available zinc capacity, because of the imbalance in the state-of-charge of the electrodes of opposite polarities. However, after charging and purposely overcharging the cells to let the zinc catch up with the silver, the capacity was restored in the range of 5 to 6 Ah (Table XXX).

Attempts were made to reduce the gassing which was deemed excessively abnormal. It was established on our internal program that applying a layer of 1 mil of inorganic material 3420-25 on the 3420-09 rigid separator side facing the zinc electrode reduces gas evolution significantly. Three full cells were then built and tested on the 2-hour period regime to quantitatively determine the amount of gassing reduction.

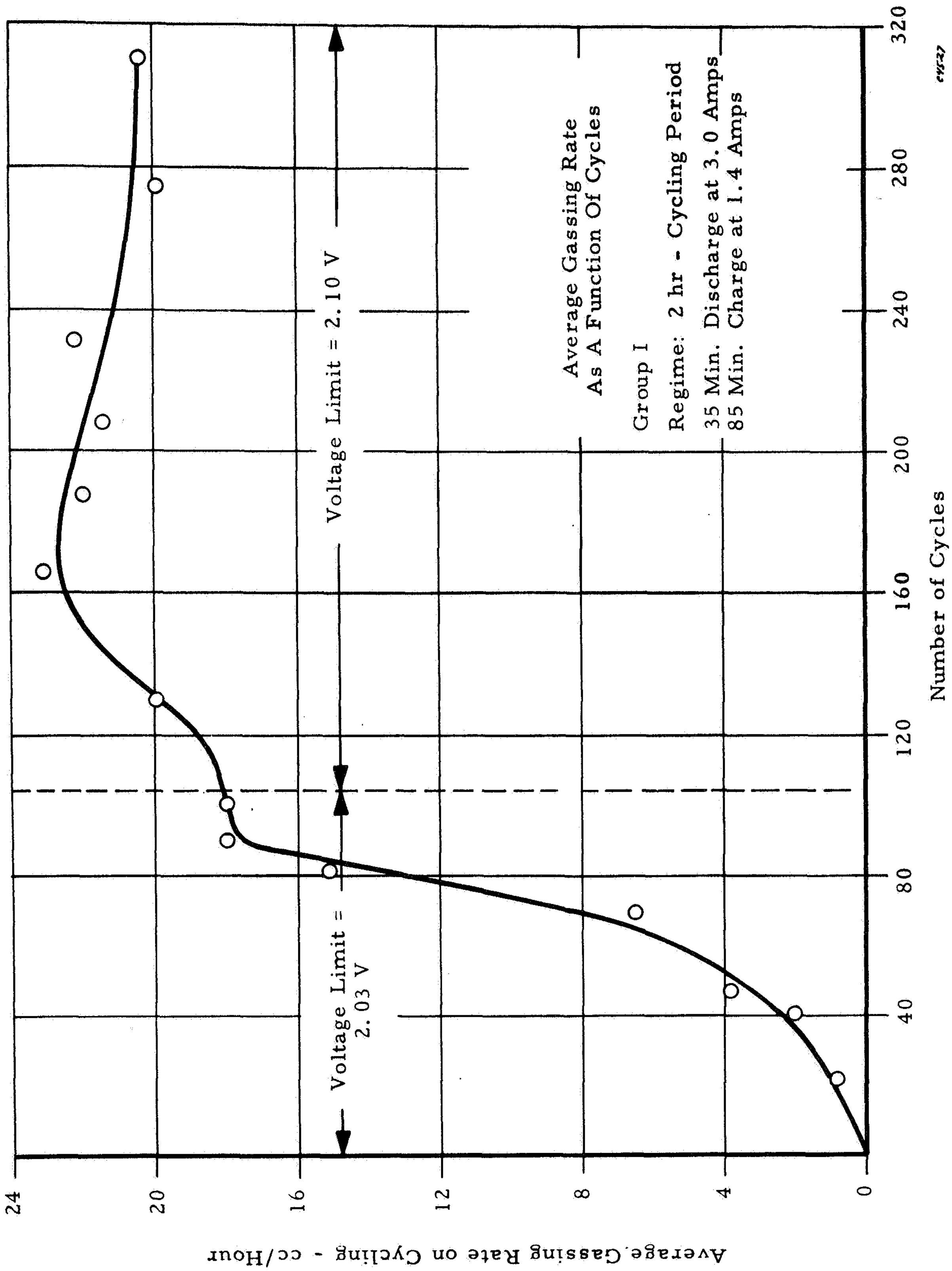


Figure 23. Average Gassing Rate of Free Venting Cells (DA-5) on Cycling at 25°C

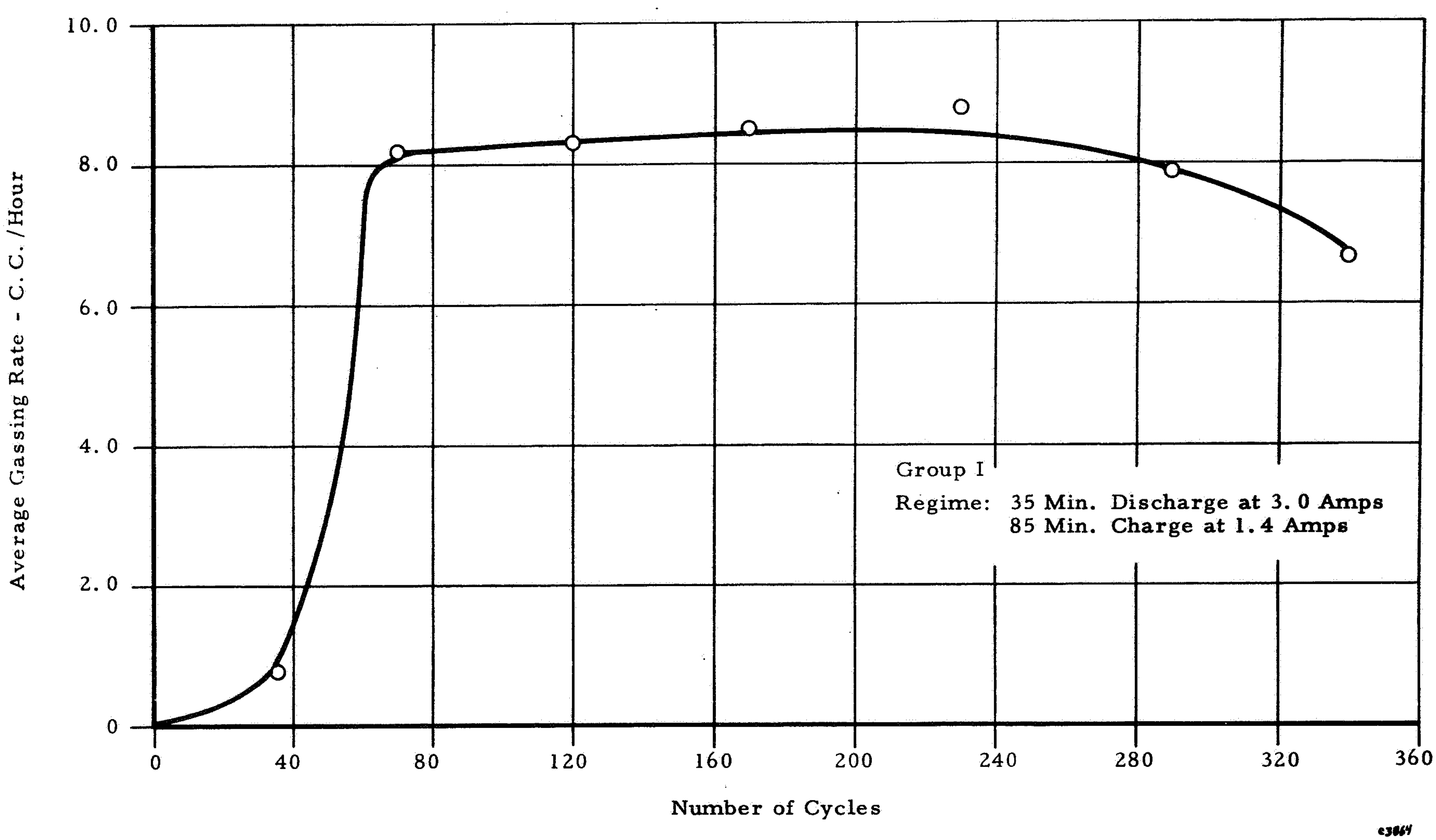


Figure 24. Average Open Circuit Gas Evolution of Free Venting Cells (DA-5) After End of Charge, on Stand at 25°C

TABLE XXIX

COMPARISON OF AMOUNTS OF GAS COLLECTED AND GAS EQUIVALENT OF ELECTROLYTE CONSUMED DURING THE ENTIRE TESTING PERIOD

Group I

Cycling Regime:	3 A discharge for 35 minutes, 1.4 A charge for 85 minutes.
Voltage Limit:	2.10 V per cell.
Temperature:	25°C
Total Cycles:	360
Total Wet Life:	80 days.

Set-Up	Cell No.	Water Weight Loss	Moles Water	Stoichiometric Gas Equivalent (ℓ)	Gas Evolved (ℓ) Measured
Free-Venting	1	13 g	0.712	24.0	19.4 ±5%
	2	14	0.777	26.1	21.2
	3	12	0.666	21.7	21.6
	4	13	0.712	24.0	21.3
	5	13	0.712	24.0	16.9
Average:		13	0.712	24.0	20.0
Pressurized	6	14	0.777	26.1	15.5 ±5%
	7	15	0.835	28.0	16.1
	8	15	0.835	28.0	(8.7*)
	9	14	0.777	26.1	18.7
	10	15	0.835	28.0	17.9
Average		14.5	0.806	27.0	17.0

*System leaked, value eliminated from average.

TABLE XXX

TESTS RUN AFTER COMPLETION OF 360 CYCLE GASSING TEST

Cell No.	Output After Cycling			Input to 2.15 V	20 Hours OCV > 1.85 V Output		
	at 3 A	at 0.5 A	Total		3 A	0.5 A	Total
1	2.25	1.15	3.4 Ah	9.2 Ah	6.0	0	6.0
2	1.50	0	1.5	6.4	3.8	0	3.8
3	1.65	0	1.7	1.9	1.0	0	1.0
4	2.25	3.35	5.6	4.9	4.0	0	4.0
5	0.25	2.90	3.15	4.3	0.75	2.1	2.85
6	2.00	0.25	2.25	6.4	4.5	0	4.5
7	2.25	0.10	2.35	6.4	3.9	0	3.9
8	1.50	0.4	1.9	5.4	3.3	0	3.3
9	1.5	0	1.5	5.4	3.0	0	3.0
10	1.5	0	1.5	4.8	3.0	0	3.0

After charge to 2.10 V with a minimum input of 5 Ah regardless of voltage, and a maximum of 9 Ah.

Cell No.	Input (Ah)	End Voltage	OCV After 17 Hours	Output			Plateau Voltage at 3 A
				3 A	0.5 A	Total	
1	8.4	2.10	1.87	4.75	1.25	6.0	1.36
2	8.4	2.09	"	5.5	0.5	6.0	1.37
3	8.4	2.08	"	5.0	1.0	6.0	1.36
4	8.4	2.12	"	5.25	1.4	6.25	1.28
5	5.25	2.18	"	0.75	4.0	4.75	1.20
6	8.4	2.09	"	4.25	0.75	5.0	1.34
7	8.4	2.11	"	5.25	1.0	6.25	1.36
8	8.4	2.12	"	5.0	1.25	6.25	1.36
9	8.4	2.12	"	4.75	1.0	5.75	1.37
10	8.4	2.13	"	5.0	1.4	6.4	1.34

These cells have been removed from test after completing 241 cycles. Gas evolution was significantly lower (see Figure 25) when compared with the first 10 cell group run at 25°C over the same number of cycles. It shows a definite improvement and the present gassing may be more related to the high rate of charge of the 2-hour period regime. This is evidenced by the fact that after a few hours on stand the gassing rate is considerably reduced and tapers off, whereas the first 10 cell group exhibited constant gassing evolution in the same period (Figure 26).

The gas composition was almost entirely hydrogen as shown below.

Cell No.	Cycle No.	
	96	107
2	97% H_2	90% H_2
3	100% H_2	95% H_2

At that time, the cells were built with large size positive electrodes which, as noted in paragraph 3.1, led to premature zinc limiting conditions and may account for the predominant hydrogen composition of the gas evolved. In order to eliminate this factor, these cells were replaced by two new 3-cell groups with the regular (smaller) silver electrodes on the 2-hour regime to confirm cycling and gassing behavior compared to the original 10-cell group I. Formation data are given in Table XXXI.

Two new series of three cells each were started on the 2-hour period cycling regime for gassing rate determination. A definite improvement in the reduction of the gassing rate was observed. This was obtained as the result of several factors — purification of the basic ingredients of the inorganic separator, maintenance of the limiting voltage on charge at a low level (2.03 V/cell average) and change of the electrolyte concentration which was raised to 40% in anticipation of the wet stand tests. The gassing test data are depicted in Figures 27 and 28 and compiled in Table XXXII.

Over 360 cycles (30 days cycling on the 2-hour regime), the gassing rate averaged about 1 cc/hour, whereas the total gas volume evolved is equivalent to less than 0.5 cc of electrolyte. No replenishment of the cells was done

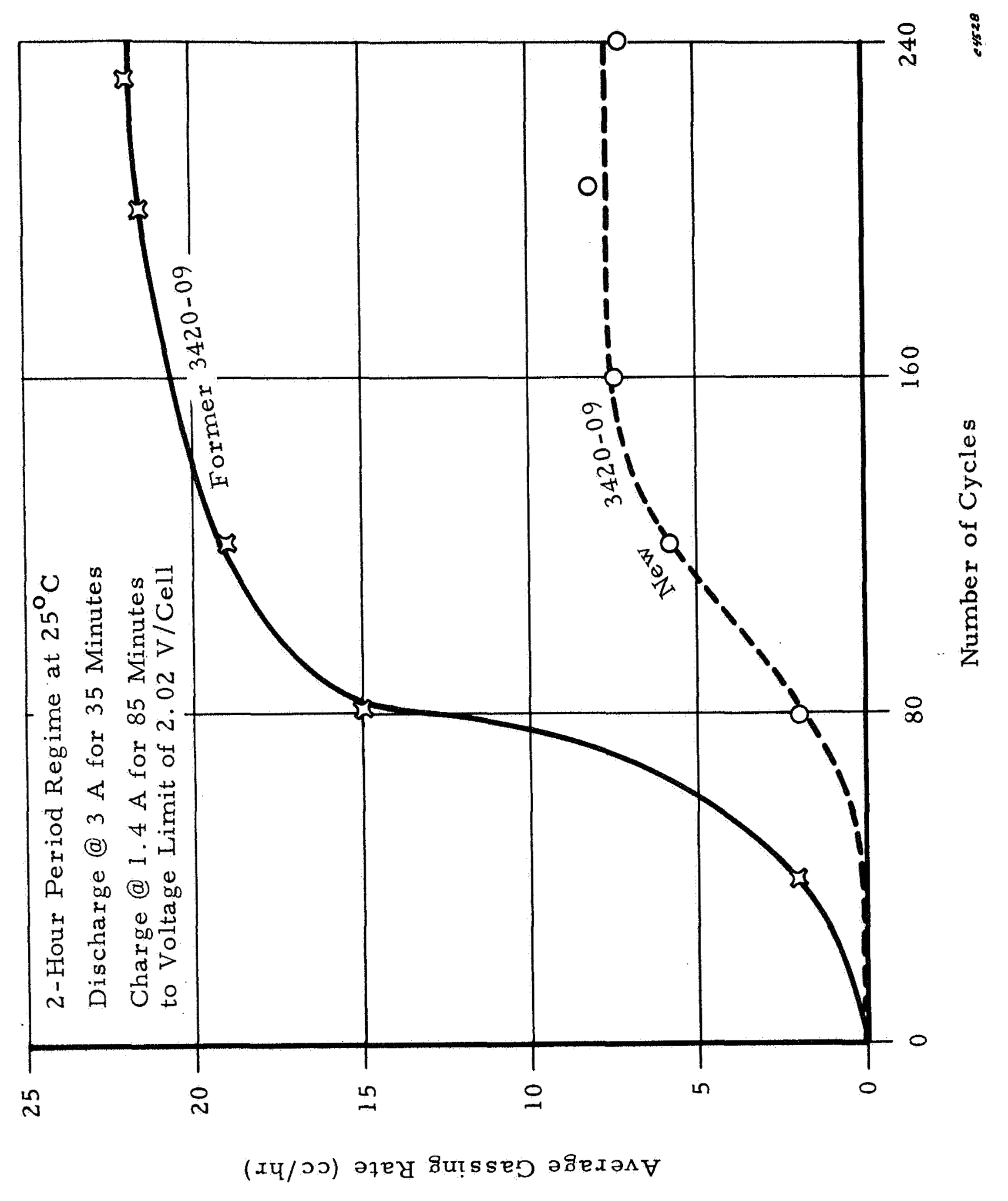


Figure 25. Gassing Rate of Cells on Cycling

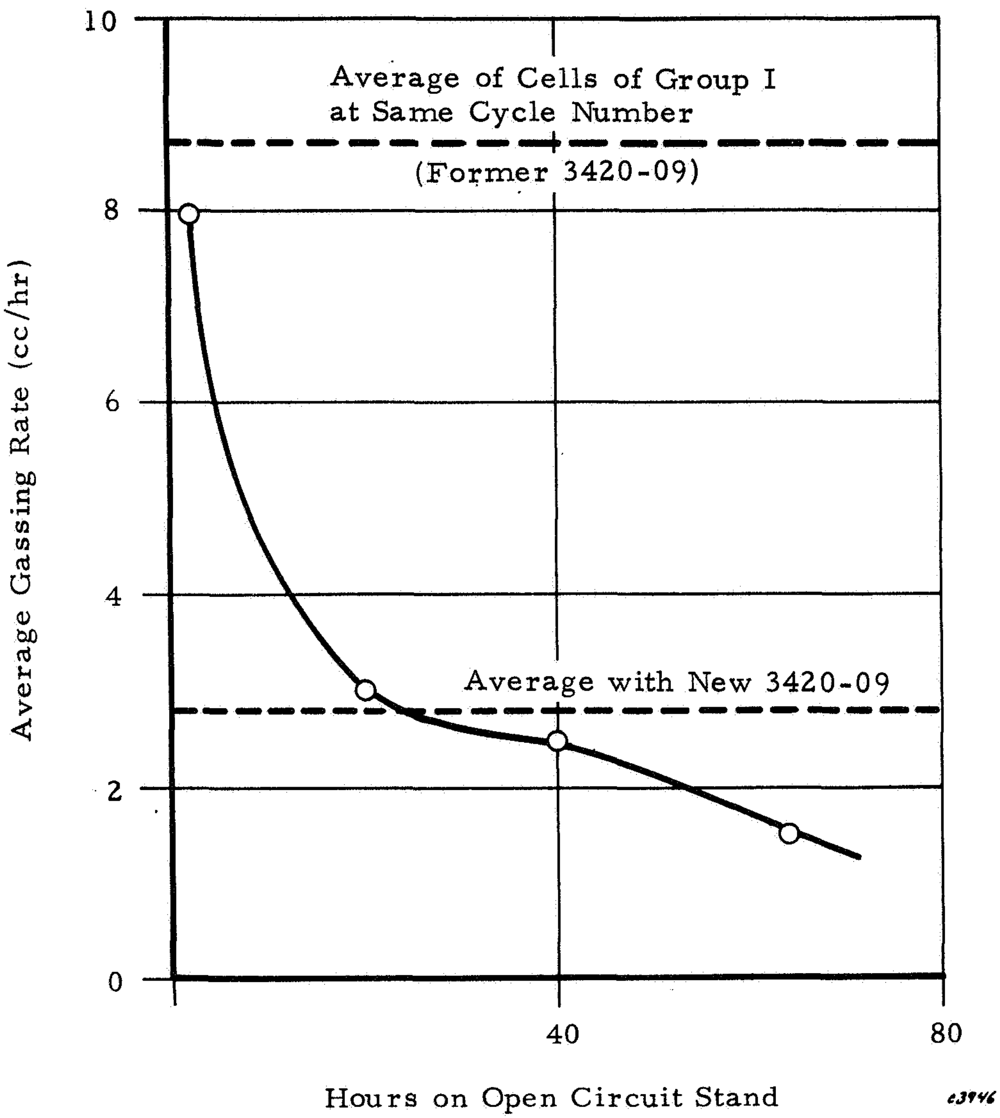


Figure 26. Gassing Rate of Cells on Open Circuit Stand at 25°C (after 215 cycles on 2-hour regime)

TABLE XXXI

FORMATION OF CELLS WITH NEW 3420-09

(Discharge at 1.0 A to 1.0 V)

Group	Cell No.	Capacity A	Voltage
ZL-33	ZL-33-1	8.0 Ah	1.44 V
	ZL-33-2	8.2 Ah	1.44 V
	ZL-33-3	7.7 Ah	1.44 V
	Average	8.0 Ah	1.44 V
ZL-34	ZL-34-1	7.8 Ah	1.45 V
	ZL-34-2	7.7 Ah	1.45 V
	ZL-34-3	7.7 Ah	1.45 V
	Average	7.7 Ah	1.45 V

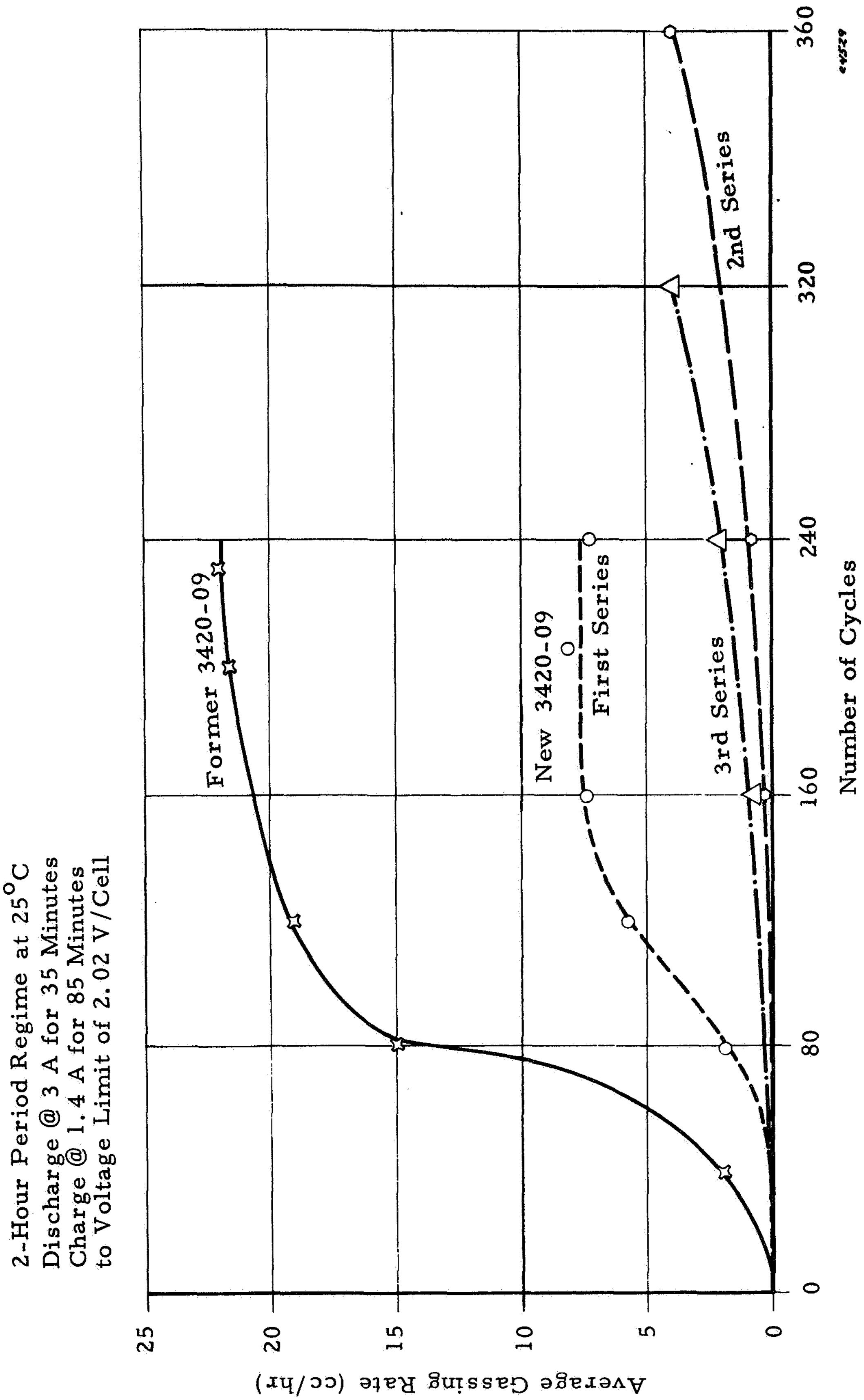


Figure 27. Gassing Rate of Cells on Cycling

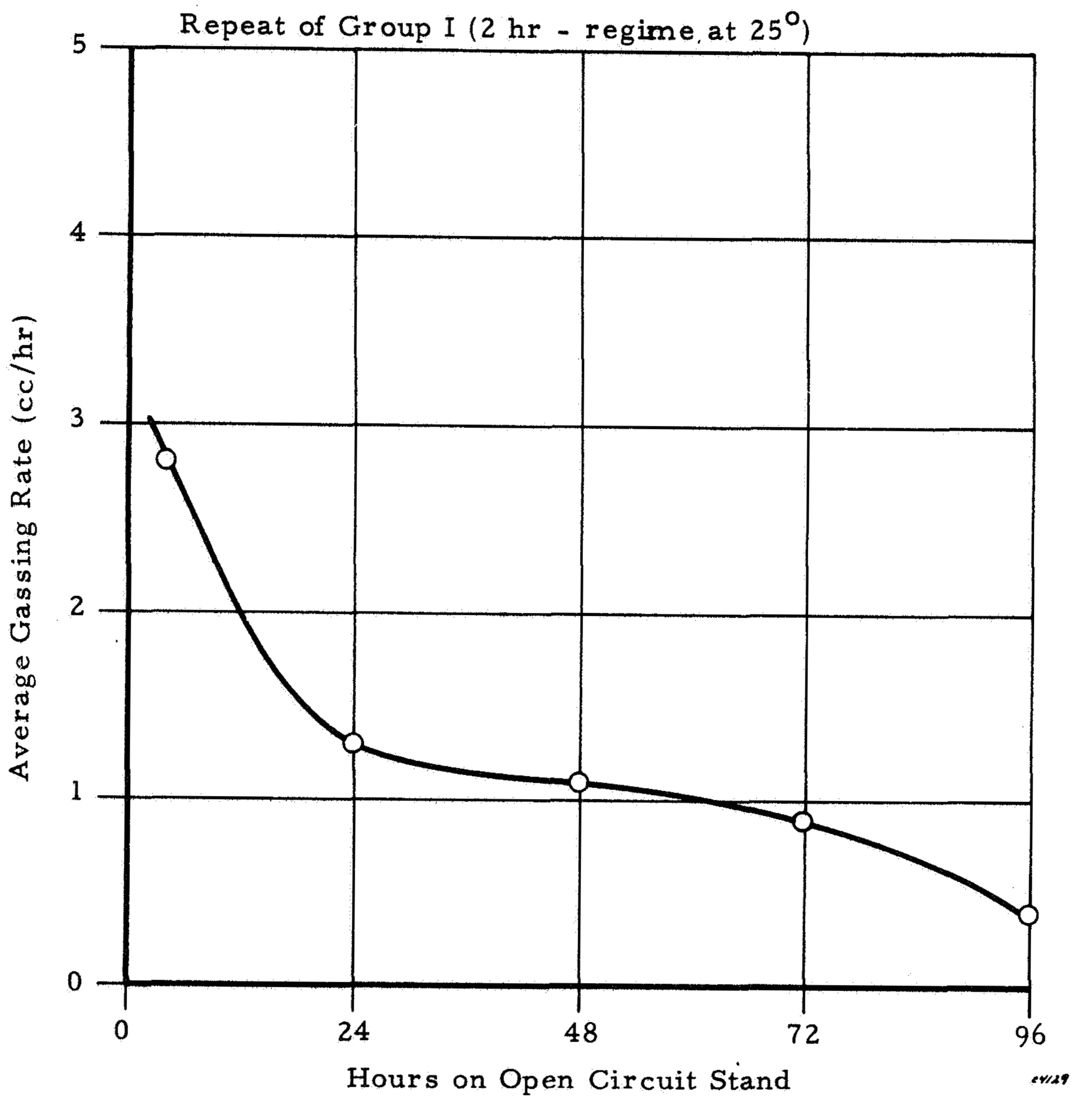


Figure 28. Gassing Rate of Cells on Open Circuit Stand at 25°C (After 326 Cycles)

TABLE XXXII

GASSING TESTS

Group I-A:
(Repeat of Group 1 with new 3420-09)

Temperature: 25°C
Cycling Period: 2 hours
Discharge: 3 A for 35 minutes
Charge: 1.6 A for 85 minutes
(voltage limited to 2.04 V/cell)

Average Data as a Function of Cycles

Time Elapsed (hrs)	Cycle No.	Free Venting Cells				
		Gas Composition			Gassing Rate (cc/hr)	Cumulative Gas Volume (cc)
		H_2 (%)	O_2 (%)	N_2 (%)		
0	0	0	82	18	—	—
24	12				0.07	2
48	24				0.18	
72	36				0.18	
96	48				0.10	
120	60				0.19	17
144	72				0.10	
168	84				0.10	
	96				0.10	
	108				0.13	
	120	14.7	70	15.3	0.11	28
	132				0.11	
	144				0.13	
	156				0.14	
336	168				0.33	44
	180				0.41	
	192				0.82	79
	216	24.4	63	12.6	0.34	
	228				0.30	131
	240				0.31	
504	252				0.42	
	276				0.76	
	288				0.88	191
	300				1.67	
	312	52	34	14	1.88	
	324				2.50	446
672	336				3.01	
	348				3.27	
720	360				4.00	695

during the entire test. However, at one point of the test, it was necessary to raise the limiting voltage to 2.05 V/cell average in order to store enough capacity in the cells so as to keep them cycling continuously.

3.1.10.2 Group II (25°C, 24-hour period)

The second group of ten cells completed one month testing on the 24-hour cycle regime. Data are presented in Table XXXIII, Figure 29 shows the variation in average gassing rate with the number of cycles. Again, the amount of gas collected from the pressurized cells was less than from the free venting cells. The hydrogen component in the gas gradually increased during the test. A period of overcharge on cycle No. 7 caused generation of oxygen which could be readily detected in the gas analysis on the 8th cycle. The gas composition during a charge-discharge cycle is given in Table XXXIV. It would appear that the oxygen content reaches a maximum value near the end of charge. Gassing rates and composition of each free venting cell are given in Table XXXV for a typical cycle.

The average gassing rate during two cycles (Nos. 30 and 31) is shown in Figure 30. Gas composition during the cycle is also indicated. The gas composition represents an average of the gas being evolved. The gassing rate rises near the end of the charge cycle and then drops to the value observed at the end of the discharge cycle. Cycling curves of free venting and pressurized cells on the third and 30th cycle are presented in Figures 31 and 32. No electrolyte was added during these tests.

At the end of 30 cycles, all cells were charged and placed on open circuit stand and the gassing rate and voltage recorded. Gas evolution on open circuit stand decreased abruptly during the first two days from 9 cc/hour to 2.8 cc/hour, then decreased to 2 cc/hour over the next 12 days (Figure 33).

The gassing rate on cycling and on OCV after cycling depends on the duration of the test and on the number of cycles, i.e., on the cycling period selected. Because of the possibility of maintaining a low voltage limit at the end of the charge, the longer cycling period is favored.

TABLE XXXIII

GASSING TESTS

Group II: Room temperature
3 A discharge for 1.2 Hours
0.17 A charge for 22.8 Hours
Average voltage limit 2.02 V/Cell

AVERAGE DATA AS FUNCTION OF CYCLES

Time Elapsed (Hours)	Cycle No.	Free Venting Cells					Pressurized Cells				
		Gas Composition			Gassing Rate (cc/hr)	Cumulative Gas Volume (cc)	Gas Composition			Pressure at end of Charge (psig)	Cumulative Vented Gas (cc)
		N_2 %	O_2 %	H_2 %			N_2 %	O_2 %	H_2 %		
24	1	30	53	18	0	0	44	32	24	0	0
120	5	22	53	27	0.8	51	19	73	6	38	9
192	8*	24	71	7	0.9	158	6	67	31	37	96
240	10	–	–	–	0.9	184	–	–	–	35	124
360	15	–	–	–	1.5	373	–	–	–	38	260
480	20	31	41	29	2.7	621	5	40	59	39	415
600	25	–	–	–	5.6	1275	–	–	–	39	750
668	27	23	20	57	8.7	1920	1	32	68	39	1282
696	29	–	–	–	10.7	2152	–	–	–	39	1703

* Average cell voltage rose to 2.08 V/cell on previous cycle by accident.

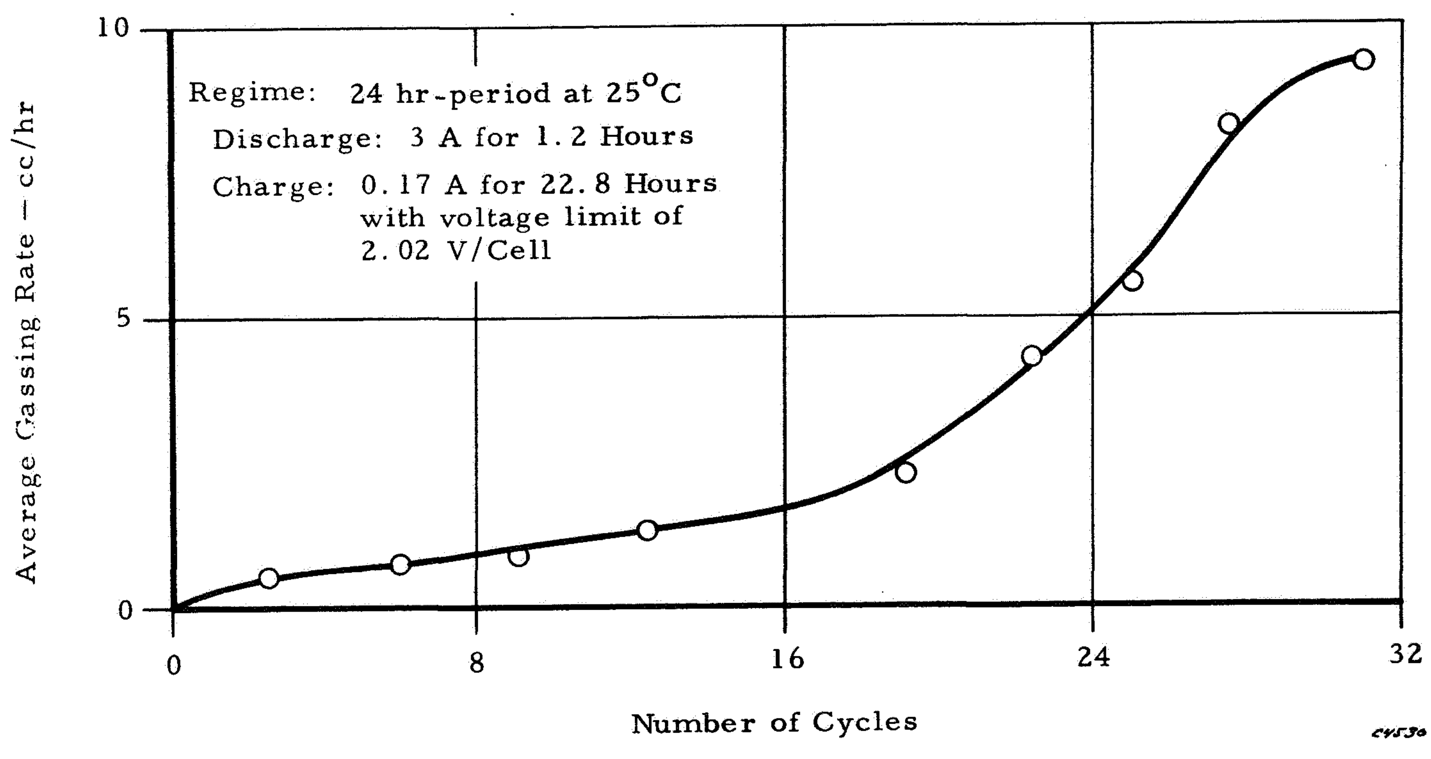


Figure 29. Average Gassing Rate of Free Venting Cells (DA-5) on Cycling

TABLE XXXIV

OXYGEN EVOLUTION DURING THE COURSE OF A CYCLE

(Group II - 24 Hour - Regime at 25°C)
(Remainder = Hydrogen)

Cell Condition	Cycle 21		Cycle 28	
	Free Venting	Pressurized	Free Venting	Pressurized
Discharge				
After 1/2 Hour	43%	43%	—	—
After 1 Hour	42%	50%	17%	19%
Charge				
After 5 Hours	17%	38%	5%	9%
After 21 Hours	38%	30%	23%	16%

TABLE XXXV

GAS COMPOSITION AND GASSING RATE OF FREE VENTING CELLS ON 27th CYCLE

(Group II - 24 Hour - Regime at 25°C)

Cell No.	Gas Composition			Gassing Rate cc/hr	Cumulative Gas Volume cc
	N_2 %	O_2 %	H_2 %		
1	26	20	54	2.1	1030
2	24	12	64	10.8	1715
3	—	—	—	3.5	1085
4	11	21	68	13.3	2285
5	30	28	42	10.0	2010
Avg.	23	20	57	7.7	1630

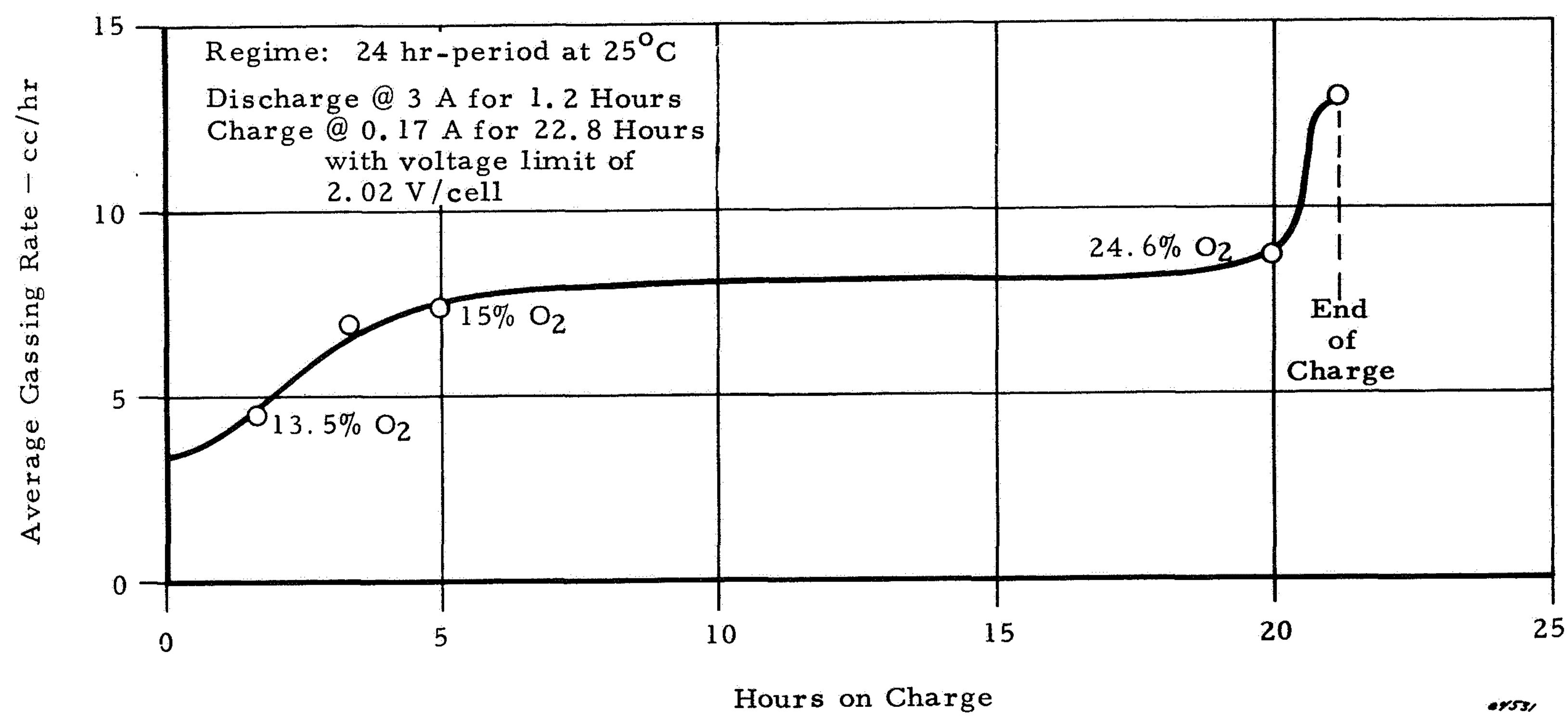


Figure 30. Average Gassing Rate and Gas Composition of Free Venting Cells on a 24-Hour Cycle (Cycle No. 30 & 31)

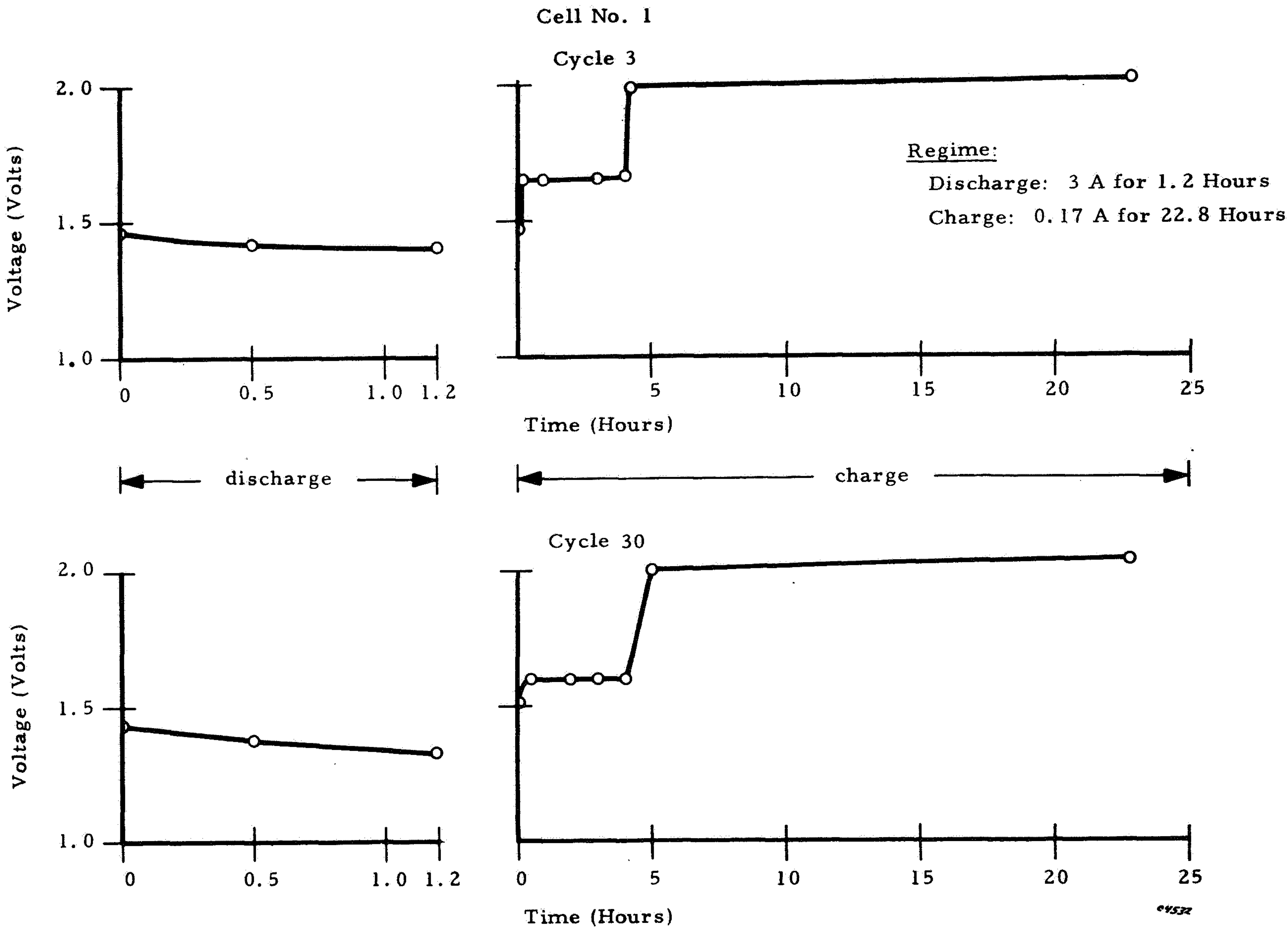


Figure 31. Cycling Curve of Free Venting DA-5 Cell on 24-Hour Regime

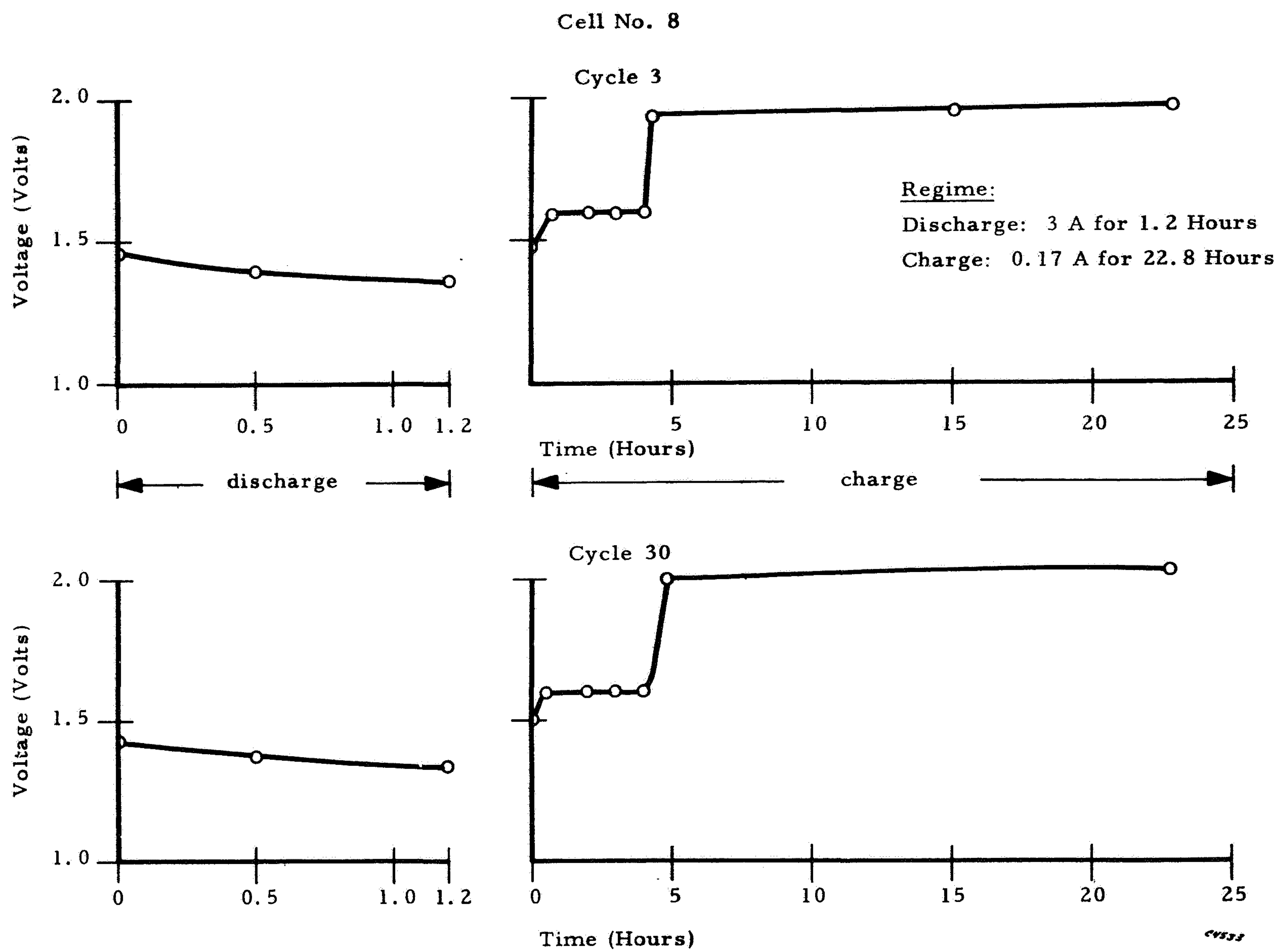


Figure 32. Cycling Curve of Pressurized DA-5 Cells on 24-Hour Regime

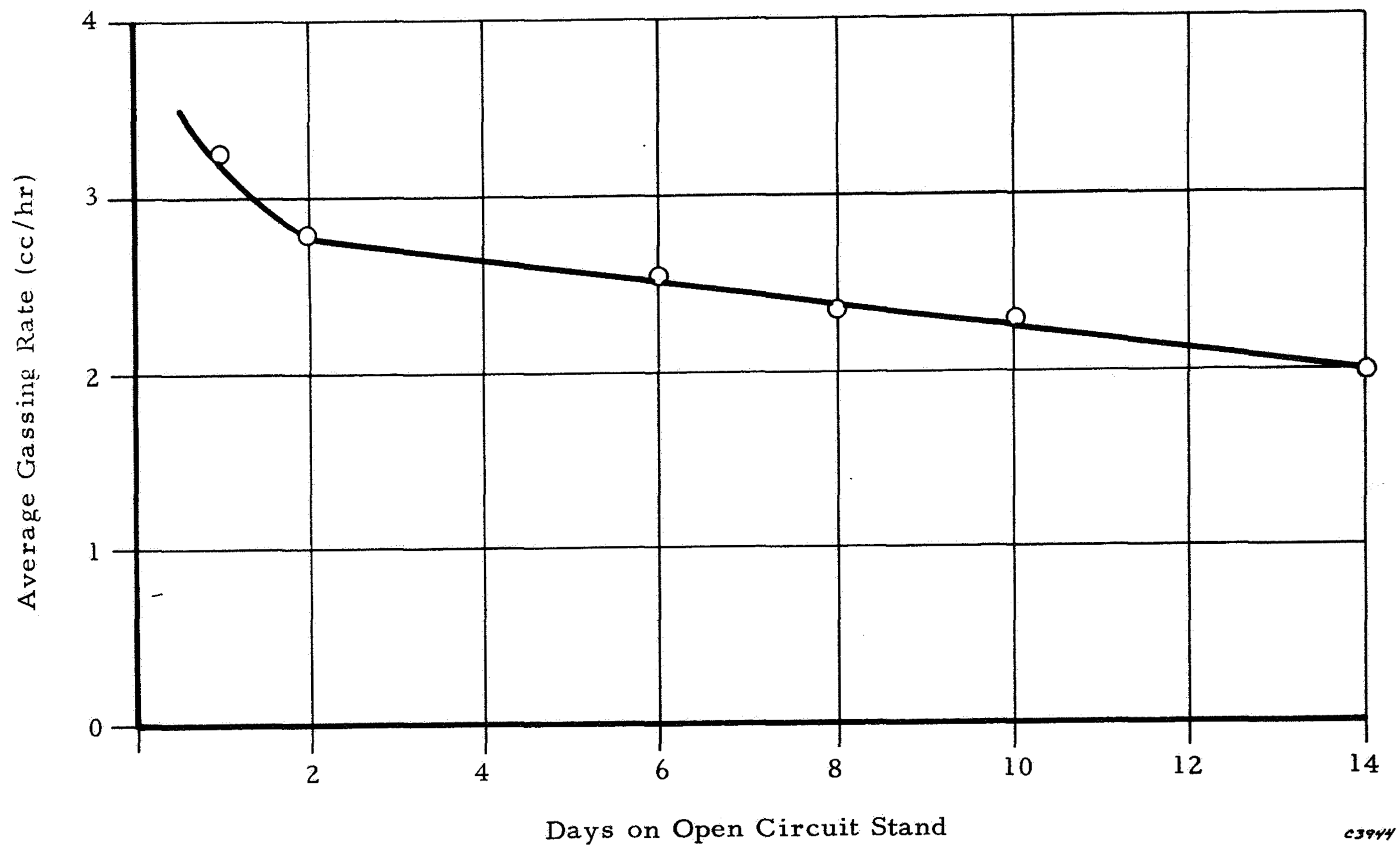


Figure 33. Average Gassing Rate of Cells on Open Circuit Stand (After 30 Cycles on a 24-Hour Regime)

3.1.10.3 Group III (40°C, 2-hour period)

The last test of the gassing study, cycling on the 2-hour period regime at 40°C was started on the following regime: 3 A discharge for 35 minutes, 1.6 A charge for 85 minutes with a voltage limit of 2.01 V/cell average.

Table XXXVI gives the complete test results at 40°C. Figure 34 gives a graphic comparison between the 25°C and 40°C tests on the same cycling regime (2-hour period), as a function of cycles over the 30-day period.

There is slightly more gassing at 40°C than at 25°C, and the difference should be considered as negligible and within normal limits of variations.

More significant is the fact that pressurized cells performed generally better than the free-venting cells; they vented less gas (831 cm^3 compared with 971 cm^3 for the free-venting cells) and their residual capacities at the end of charge after 360 cycles of 2-hour period regime were higher by an average of 40% (see Table XXXVII). However, all cells, after a normal recharge, yielded approximately 5 Ah.

3.2 TASK II: FINALIZED DESIGN, PRELIMINARY CELL TESTING

The objective of Task II is to fabricate a certain number of cells of the proposed final design for testing — charged wet stand at 25°C and 100°C, environmental tests, and electrical tests.

3.2.1 Wet Stand

Sixteen cells were scheduled for the wet stand tests as follows:

Stand Time	25°C	100°C
1 month	2 cells	2 cells
2 months	2 cells	2 cells
3 months	4 cells	4 cells
Constraint: OCV lower limit:	1.80 V	1.62 V

TABLE XXXVI

GASSING TESTS

Group III: Temperature: 40°C
Cycling Period: 2 hours
Discharge: 3.0 A for 35 minutes
Charge: 1.6 A for 85 minutes (voltage limited to 2.01 V/cell)

AVERAGE DATA AS A FUNCTION OF CYCLES

Time Elapsed (hrs)	Cycle No.	Free Venting Cells: Gas Composition H_2 (%)	Free Venting Cells: Gas Composition O_2 (%)	Free Venting Cells: Gas Composition N_2 (%)	Free Venting Cells: Gassing Rate (cc/hr)	Free Venting Cells: Cumulative Gas Volume (cc)	Pressurized Cells: Gas Composition H_2 (%)	Pressurized Cells: Gas Composition O_2 (%)	Pressurized Cells: Gas Composition N_2 (%)	Pressurized Cells: Pressure at End of Charge (psig)	Pressurized Cells: Cumulative Vented Gas (cc)
0	0	0	81.8	18.2	0	0	—	—	—	-4	0
24	12	8.4	51.8	39.8	0.59	14.2	11.6	75.0	—	7	0
48	24	—	—	—	0.56	27.6	—	—	—	15	0
72	36	32.0	48.5	19.5	0.47	39.0	28.2	63.4	8.4	24	0
96	48	—	—	—	0.45	49.8	—	—	—	28	0
120	60	—	—	—	0.44	60.4	—	—	—	35	15
144	72	33.0	45.5	21.4	0.54	74.0	34.1	54.9	7.7	30	19
168	84	—	—	—	0.62	88.8	—	—	—	31	24
	96	—	—	—	0.57	103.0	—	—	—	30	35
	108	—	—	—	0.58	117.0	—	—	—	29	48
	120	32.1	46.7	21.1	0.63	132.0	45.3	49.8	5.0	29	62
	132	—	—	—	0.63	147.0	—	—	—	30	74
	144	—	—	—	0.68	163.0	—	—	—	30	88
	156	37.1	42.3	2.05	0.66	180.0	49.0	47.5	3.5	30	104
336	168	—	—	—	0.68	196.0	—	—	—	30	116
	180	—	—	—	0.67	212.0	—	—	—	30	129
	192	44.9	35.2	19.9	0.73	230.0	55.0	41.8	3.2	30	144
	204	—	—	—	0.91	252.0	—	—	—	30	159
	216	—	—	—	0.89	273.0	—	—	—	30	177
	228	—	—	—	0.96	296.0	—	—	—	30	198
	240	61.8	22.1	16.1	0.93	318.0	68.8	26.8	4.4	30	219
504	252	—	—	—	1.29	349.0	—	—	—	30	255
	264	—	—	—	1.36	382.0	—	—	—	30	277
	276	64.7	16.3	18.9	1.70	423.0	78.0	16.6	5.4	29	318
	288	—	—	—	1.98	470.0	—	—	—	30	361
	300	—	—	—	2.39	528.0	—	—	—	30	415
	312	—	—	—	2.97	599.0	—	—	—	30	477
	324	—	—	—	—	700.0	81.8	14.1	4.1	30	548
672	336	—	—	—	3.25	802.0	—	—	—	31	631
	348	—	—	—	3.49	886.0	—	—	—	31	736
720	360	70.3	6.0	19.7	3.80	971.0	88.2	8.0	3.8	31	831

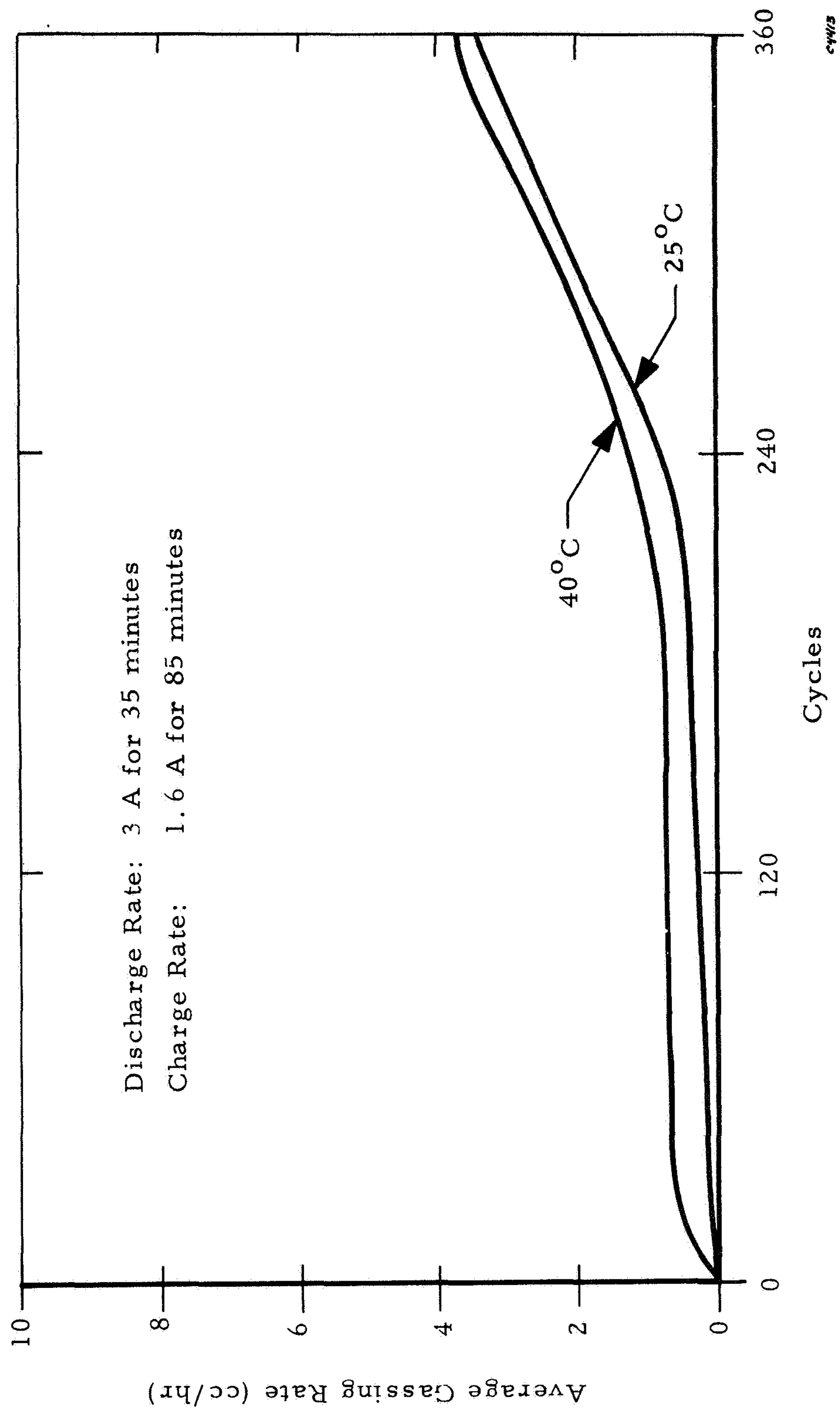


Figure 34. Gassing Rates at 25°C and 40°C on the 2 Hour-Period Regime

TABLEXXXVII

STATUS OF CELLS AFTER GASSING TEST AT 40°C

(2-Hour Period Regime)
Group III

Set-Up	Cell No.	Output After End of Charge After 360 Cycles	Output After Recharge
Free Venting Cells	ZL-43-1	3.25 Ah	5.30 Ah
	-2	2.50	5.20
	-3	2.50	5.00
	-4	2.50	5.10
	-5	2.65	4.90
	Average	2.70 Ah	5.1 Ah
Pressurized Cells	ZL-43-6	4.0 Ah	4.9 Ah
	-7	2.7	4.6
	-8	3.0	4.9
	-9	4.6	5.0
	-10	4.6	5.1
	Average	3.8 Ah	4.9 Ah

If the OCV is reached before the requisite time, the cell is to be removed from stand and discharged to determine its capacity retention (or residual capacity). The cell is then given another charge and discharge to determine the actual capacity at the time of the test.

3.2.1.1 Wet Stand at 100°C

The results reported in Tables XXXVIII and XXXIX show that the capacity loss (about 23%) is contributed by the thermal decomposition of the argentic silver oxide, when comparing residual capacity with actual capacity at the time of the test.

Comparing this with the original capacity (formation), there is a greater loss, which is due partly to the argentic silver oxide thermal decomposition and partly to the zinc degradation resulting in hydrogen evolution (faster at high temperature). The relative loss percentages are respectively 23% caused by the silver and 21% caused by the zinc. Part of this zinc loss is recovered when the cell is overcharged beyond the normal cut-off voltage of 2.05 V. This is evidenced by the two cells (#9 and #10) which were purposely charged to 2.10 V and gave an output of 6.8 Ah instead of 6.0 Ah yielded by the other cells. This reduces the capacity loss due to zinc to only 13%.

The OCV decay at 100°C (given in graphical form for all cells in Figure 35) exhibits the same pattern as the voltage decline under electrochemical conversion at a light discharge rate.

3.2.1.2 Wet Stand at 25°C

The results are reported in Table XL. Figure 36 summarizes the capacity variation with time over the 3-month period, as required by the work statement.

3.2.1.3 Extra Tests

When the tests reported in 3.2.1.1 and 3.2.1.2 were completed, the 16 cells were used for some extra tests combining cycling and charged stands over 12 months.

TABLE XXXVIII

100°C WET STAND TEST DATA

(ZL-35 Series)

Cell Number →	9	10	11	12	13	14	15	16	
<u>1st Discharge</u> (Formation)(Ah)	7.85	7.95	8.00	7.80	8.00	8.00	7.85	7.85	Q_1
First Run									
OCV at start (V)	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	
OCV after:									
21 hrs 100°C	1.58	1.56	1.59	1.59	1.59	1.56	1.58	1.58	
42 hrs 100°C	1.57	1.56	1.58	1.58	1.58	1.56	1.58	1.58	
<u>2nd Discharge (Ah)</u>	4.05	4.10	4.65	4.50	4.25	4.00	4.50	4.70	Q_2
Recharge: Input to 2.05 V	5.5	5.5	6.1	5.5	5.5	6.3	6.3	6.3	
<u>3rd Discharge (Ah)</u>	5.1	5.0	6.0	5.25	5.30	6.25	6.25	6.25	Q_3
Recharge	—	—	—	—	—	—	—	—	
Second Run									
OCV at start	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	
OCV after periods of:									
9 hrs 100°C	1.85	1.85	1.85	1.85	1.84	1.83	1.83	1.84	
9 hrs 100°C	1.85	1.84	1.81	1.81	1.70	1.64	1.69	1.73	
9 hrs 100°C	1.78	1.70	1.59	—	—	—	—	—	
9 hrs 100°C	1.60	1.58	—	—	—	—	—	—	
15 hrs 25°C	1.58	1.58	1.57	1.57	1.58	1.55	1.57	1.43	
16 hrs 25°C	1.56	1.56	—	—	—	—	—	—	
Hrs at 100°C above 1.58 V	36	36	27	27	18	18	18	18	
<u>4th Discharge (Ah)</u>	4.9	4.9	4.6	4.6	4.4	4.5	4.6	4.75	Q_4
Recharge: Input (Ah) to 2.05 V	5.7	5.6	6.75	6.75	6.1	6.1	6.1	6.1	
to 2.10 V	6.9	6.9							
<u>5th Discharge (Ah)</u>	(6.8)*	(6.8)*	6.30	6.25	6.0	5.9	5.95	5.95	Q_5

* discounted from average

TABLE XXXIX

100°C WET STAND TEST
AVERAGES OF 8 CELLS

(ZL-35 Series: Cells 9 through 16)

Tests	Consecutive actions as defined by Table II		Average Outputs (Ah)
	Formation	Q_1	7.75 Ah
First Run	Discharge after 42 hours at 100°C.	Q_2	4.35 Ah
	Normal recharge, then discharge.	Q_3	5.65 Ah
Second Run	Normal recharge, then submitted to average of 24 hours at 100°C.		
	Discharge.	Q_4	4.65 Ah
	Normal recharge, then discharge.	Q_5	6.05 Ah
	Capacity retention with respect:		
First Run	To formation.	Q_2/Q_1	56%
	To actual capacity after test.	Q_2/Q_3	77%
Second Run	To actual capacity before test.	Q_4/Q_3	82%
	To actual capacity after test.	Q_4/Q_5	77%

Weight Loss (Electrolyte): 0.14 g/hr at 100°C for all cells over their entire exposure to 100°C.

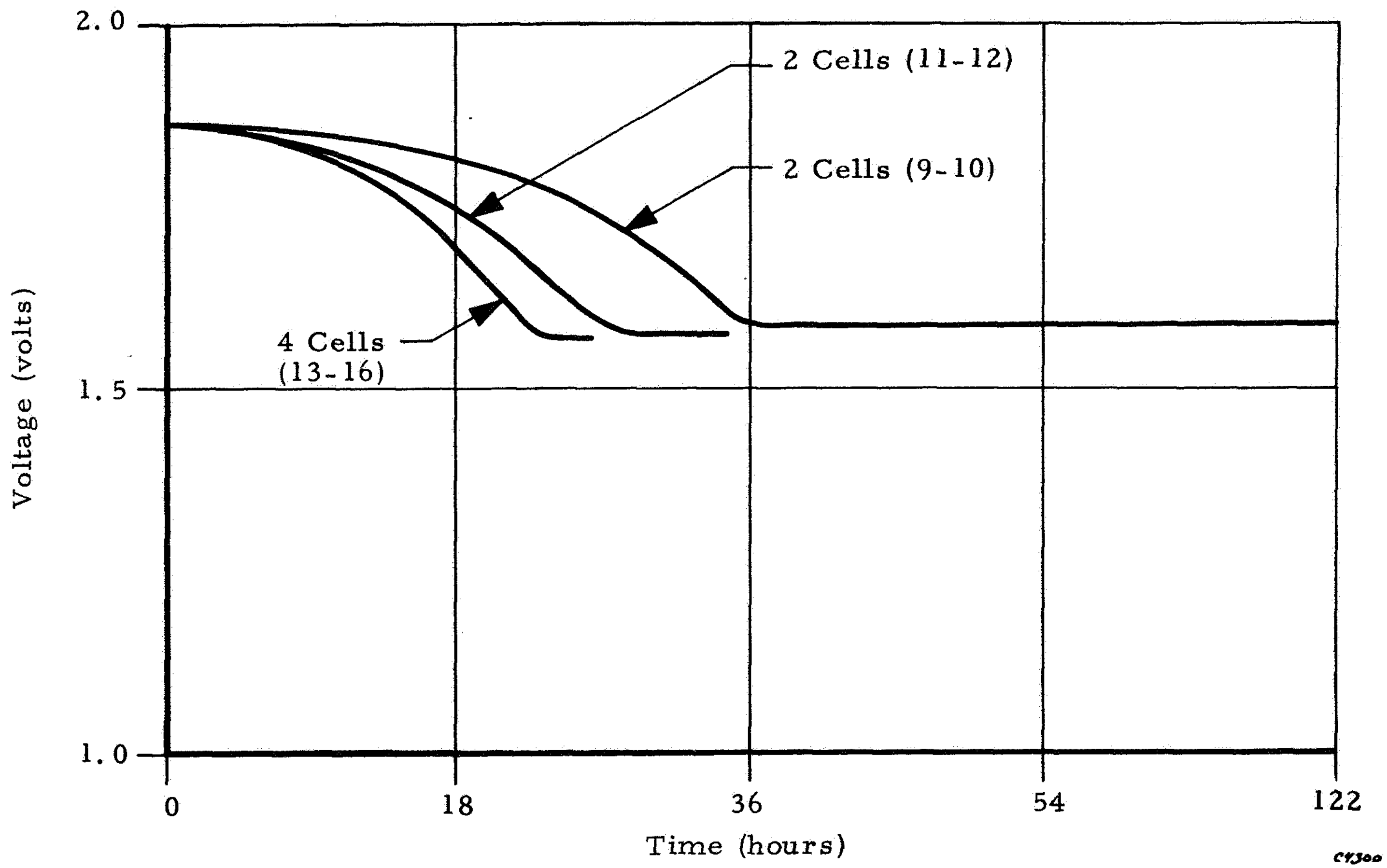


Figure 35. OCV Drop at 100°C of Charged 5 Ah-Cells (Model DA-5-1-N) (2nd run)

TABLE XL

25°C WET STAND TEST

(ZL-35 Series)

Group	I		II		III				
Cell Number	1	2	3	4	5	6	7	8	
1st Discharge (Ah) (Formation)	7.90	7.80	7.95	7.80	7.95	7.50	7.95	7.95	Q_1
OCV at Start (Volts)	1.86	1.86	1.86	1.86	1.86	1.86	1.86	1.86	
Recharge	—	—	—	—	—	—	—	—	
OCV After:									
1 month (Volts)	1.85	1.85	1.85	1.85	1.85	1.85	1.85	1.85	
2 months	—	—	1.85	1.85	1.85	1.85	1.85	1.85	
3 months	—	—	—	—	1.85	1.85	1.85	1.85	
2nd Discharge (Ah)									
1 month group	6.85	6.85	—	—	—	—	—	—	Q_{21}
2 month group	—	—	6.6	6.5	—	—	—	—	Q_{22}
3 month group	—	—	—	—	6.55	6.70	6.50	6.60	Q_{23}
Recharge	—	—	—	—	—	—	—	—	
3rd Discharge (Ah)	8.0	7.60	7.60	7.40	7.45	7.45	7.45	7.55	Q_3
Average	1 Month		2 Months		3 Months				
Capacity Retention with respect to:									
Original	88%		83%		84%				$\frac{Q_2}{Q_1}$
Actual After Test	88%		88%		88%				$\frac{Q_2}{Q_3}$

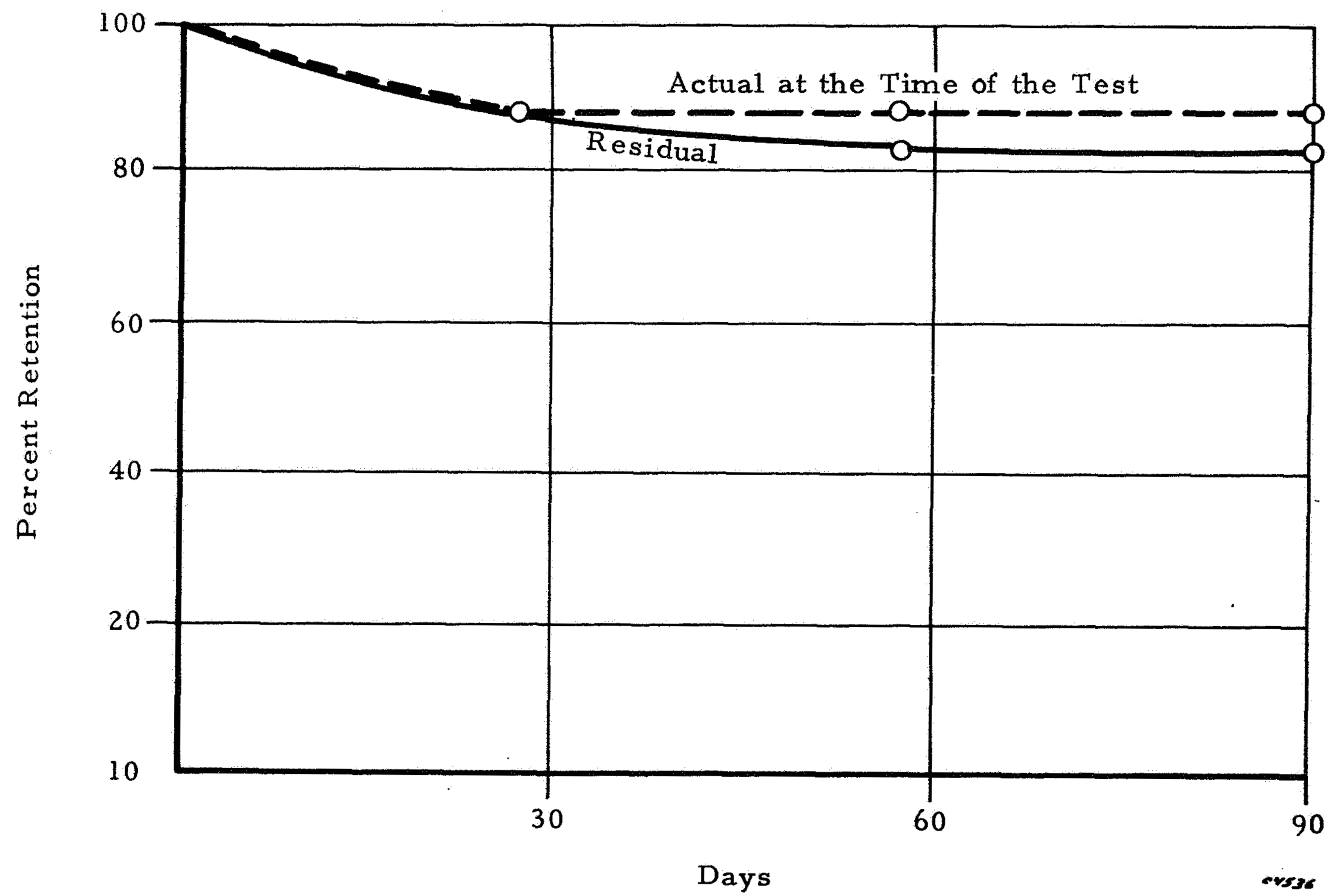


Figure 36. Capacity Retention on Charged Stand Over a 3-Month Period at 25°C

Tables XLI and XLII show the chronological tests performed on the 8 cells (#1 through #8) previously run at 25°C and the 8 cells (#9 through #16) previously run at 100°C. All had a charged wet stand period of 8 months prior to the last discharge at the end of their 12 months wet life.

All cells retained an OCV of over 1.84 volts at the end of their last stand period.

3.2.2 Environmental Tests

A group of five cells was formed, charged and submitted to the environmental tests (shock, acceleration and vibration) as defined in Task II. (See Appendix A.) The cells maintained an OCV of 1.86 V throughout the tests. No physical damage was noticed. The test report submitted by the Ogden Technology Laboratories was sent to the NASA Project Manager.

The cells were then submitted to electrical testing along with cells not environmentally tested, as described in the next paragraph.

3.2.3 Electrical Tests

Fifteen cells were scheduled for this test: five cells previously submitted to environmental testing; (as described in the previous paragraph, designated group A) and five cells used as controls (designated group B) and five cells provided with pressure gauges (designated group C).

The regime used on the cells of this group was intended to be severe in order to bring about any major deficiency on a fast preliminary evaluation in the range of 500 cycles.

The work statement calls for a regime of 1.5 hour-cycling period, 1/2-hour discharge, 1-hour charge, at 30% depth of discharge based on actual capacity. Taking 7.5 Ah as the average original actual capacity, the discharge and charge currents must therefore be 4.5 A and 2.5 A respectively.

This regime is extremely severe for this cell in three respects:

TABLE XLI

WET STAND EXTRA TESTS

(Combining wet stand and cycling over one year)

Cell No.	1	2	3	4	5	6	7	8
Cycle 1 Output	7.9 Ah	7.8 Ah	7.9 Ah	7.8 Ah	8.0 Ah	7.5 Ah	7.8 Ah	8.0 Ah
Charged wet stand at 25°C	1 month		2 months		3 months			
Cycle 2 Output	6.8 Ah	6.8 Ah	6.6 Ah	6.5 Ah	6.6 Ah	6.7 Ah	6.5 Ah	6.6 Ah
Discharged wet stand and some cycling	3 months 1 cycle		2 months 4 cycles		1 month 0 cycle			
Output before new stand	8.0 Ah	7.9 Ah	8.2 Ah	8.1 Ah	8.2 Ah	8.2 Ah	8.2 Ah	7.8 Ah
Charged wet stand at 25°C	8 months							
Output after stand	5.8 Ah	5.7 Ah	5.7 Ah	5.7 Ah	5.7 Ah	5.7 Ah	5.8 Ah	5.9 Ah

TABLE XLII

WET STAND EXTRA TESTS (OUTPUTS)

(Combining charged wet stand and cycling over one year)

Cell No.	9	10	11	12	13	14	15	16
Total 12 months	7.85 Ah	7.95 Ah	8.0 Ah	7.8 Ah	8.0 Ah	8.0 Ah	7.85 Ah	7.85 Ah
	$100^{o}C$ wet stand tests (3 cycles – 1 month)							
	6.8 Ah	6.8 Ah	6.3 Ah	6.25 Ah	6.0 Ah	5.9 Ah	5.95 Ah	5.95 Ah
	3 months – discharged condition at $25^{o}C$							
	8 months – charged condition at $25^{o}C$							
	4.4 Ah	4.7 Ah	5.3 Ah	4.6 Ah	6.3 Ah	5.1 Ah	5.2 Ah	5.3 Ah

Total wet life: 12 months

Total cycles: 6

1. The depth of discharge of 30% of actual capacity is really 45% of the rated capacity, since the cell is designed for a 5 Ah nominal capacity.

2. On the other hand, the cell is intended for a low rate operation and the 4.5 A discharge current is relatively too high for efficient performance since the plateau voltage will be low.

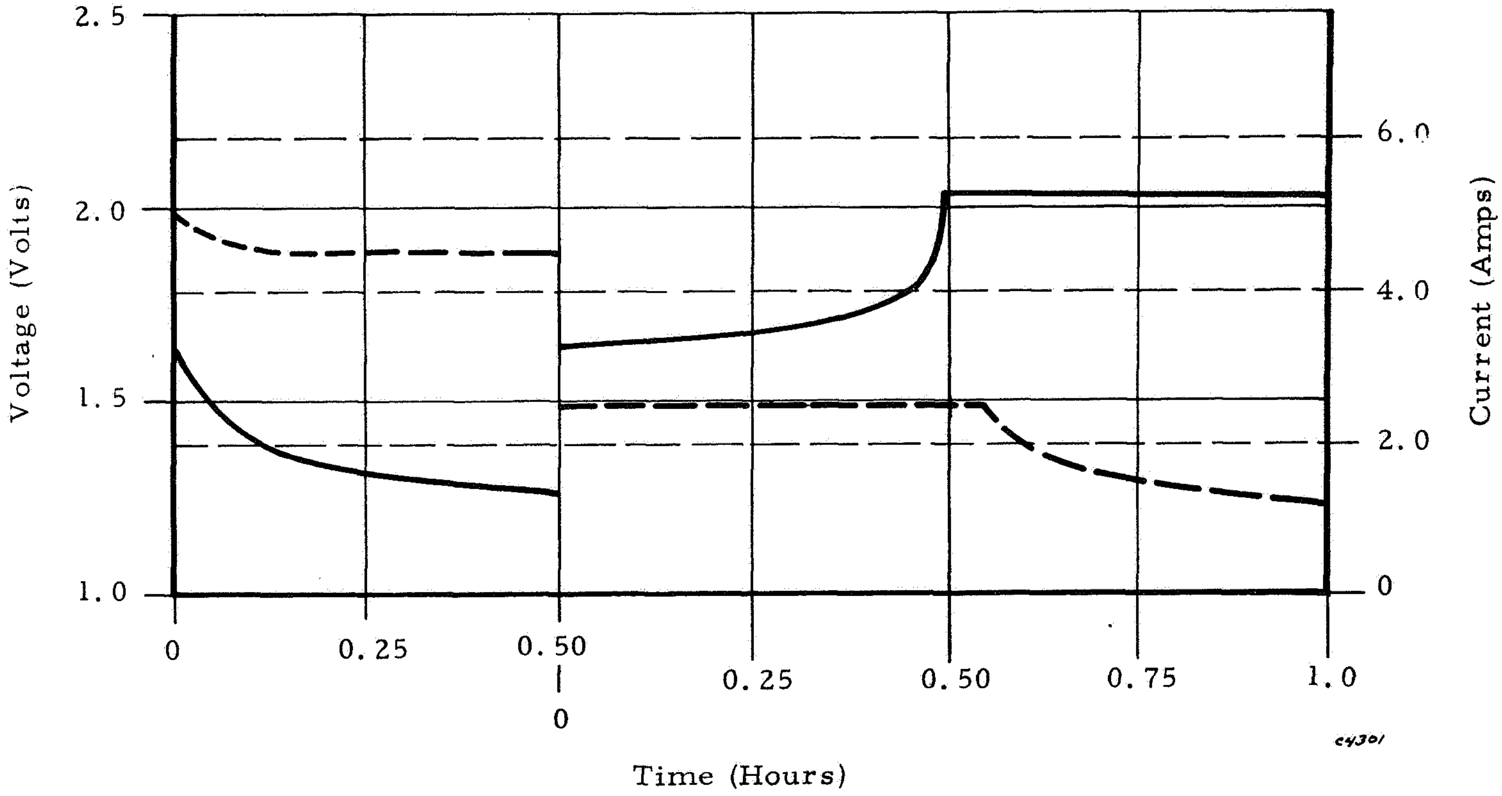
3. The cycling period imposes a recharge in one hour at a very high rate (20 mA/cm^2 instead of the normal 3 mA/cm^2) so that the coulombic efficiency is impaired and overcharges up to 25% may be needed. This has two side effects on the cell performance: heavy gassing which causes cutdown electrode surface area with subsequent higher current density and, more important, promotion of zinc penetration and zinc slumping.

The voltage limit set originally at 2.05 V/cell was found inadequate because of the high rate of charge after 170 cycles of this regime. A frequent verification of the output and input capacities show that the input was constantly lower than the output, because the high charging rate polarizes the cells very fast and raises their voltage prematurely to a point where the current tapers off to a low value. A typical voltage-current cycling curve is shown in Figure 37. The standard 2.05 V limit is therefore insufficient to maintain a reasonable current over the 1-hour charge. At cycle 170, a few cells started showing signs of coming close to the 1.0 V limit before the end of the requisite discharge time. It was decided to raise the voltage to a point where the input was higher than the output. It is a fact that the voltage limit and the coulombic efficiency are a function of the charging rate and of the temperature. The limit was raised in small increments until the cells could cycle without frequent recharges.

An extra group of five cells (Group D) was put on cycling with the high voltage limit (2.15 V/cell) set at the beginning.

Data up to 500 cycles were sent to the NASA Program Manager for review and approval to start Task III.

Cycling curves of groups A, B, and C show that the cells were at the beginning constantly undercharged.



Solid Line: Voltage
Dotted Line: Current

Figure 37. Typical Voltage-Current Relationship During Cycling

All data are presented at periodic cycles in the tables entitled "Uniformity Study" giving for each cell of each group:

1. the charge characteristics:

   OC = overcharge percentage
   m% = monoxide plateau percentage of total charge period
   V_f = final voltage at the end of the charge period.

2. the discharge characteristics:

   p% = peroxide plateau percentage of total discharge period
   V_p = plateau voltage
   Ve = end voltage at the end of the discharge period

3. the electrolyte addition in cumulative amounts from the beginning of the test.

The referenced tables and figures are:

for	Group A	Tables XLIII, XLIV, XLV	Figures 38, 39
	Group B	Tables XLVI, XLVII, XLVIII	Figures 40, 41
	Group C	Tables IL, L, LI	Figures 42, 43
	Group D	Tables LII, LIII, LIV	Figures 44, 45

Table LV is a compilation of the total cycles to failure on all cells of the four groups with their final capacity check.

The post-failure examination of the cell components showed common traits of failure for all cells of the four groups:

- A few cracked separators
- Evidence of zinc penetration
- Severe zinc electrode erosion and slumping (50 to 60%).

The wafer edge sealant was intact and the positive electrodes in good condition, although half-way discharged because of the missing zinc eroded area (see Figure 46).

3.3 TASK III: FABRICATION, TEST AND DELIVERY OF CELLS

The objective ot Task III was the fabrication of 210, 5-Ah cells for testing. The cells were fabricated in six lots of 35 cells each according to the finalized design, approved at the end of Task II. Figure 47 is a photograph of completed cells.

TABLE XLIII

UNIFORMITY STUDY, GROUP A (ENVIRONMENTALLY TESTED)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.5 A for 1.0 hr
Voltage Limit: 2.05 V/cell
Temperature: 25°C

Cycle 1-10

Cell Number		1	2	3	4	5	Avg.
Charge	m%	38	38	38	38	38	38
(OC = -8%)	V_f	2.06	2.05	2.07	2.07	2.06	2.06
Discharge	p%	19	19	19	19	19	19
	V_p	1.32	1.33	1.33	1.34	1.31	1.33
	V_e	1.28	1.28	1.29	1.29	1.28	1.28
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

Cycle 100

Charge	m%	34	34	34	34	34	34
(OC = -5%)	V_f	2.08	2.05	2.07	2.06	2.09	2.07
Discharge	p%	19	19	19	19	19	19
	V_p	1.28	1.32	1.30	1.30	1.29	1.30
	V_e	1.25	1.29	1.28	1.27	1.25	1.27
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

TABLE XLIV

UNIFORMITY STUDY, GROUP A
(ENVIRONMENTALLY TESTED)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.5 A for 1.0 hr
Voltage Limit: 2.05 V/cell
Temperature: 25°C

Cycle 170

Cell Number		1	2	3	4	5	Avg.
Charge	m%	31	41	31	31	62	39
(OC = -2%)	V_f	2.03	2.02	2.10	2.05	1.98	2.04
Discharge	p%	25	25	25	25	25	25
	V_p	1.23	1.20	1.25	1.15	1.22	1.21
	V_e	1.20	1.15	1.22	1.10	1.18	1.17
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

Cycle 310*

Charge	m%	26	26	26	26	26	26
(OC = +16%)	V_f	2.07	2.19	2.33	2.09	2.03	2.14
Discharge	p%	25	25	25	25	25	25
	V_p	1.24	1.24	1.19	1.26	1.26	1.24
	V_e	1.10	1.19	1.13	1.24	1.20	1.19
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

*After Cycle 200, voltage limit raised to 2.15 V/cell, and current to 2.75 A.

TABLE XLV

UNIFORMITY STUDY, GROUP A
(ENVIRONMENTALLY TESTED)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.75 A for 1.0 hr
Voltage Limit: 2.15 V/cell
Temperature: 25°C

Cycle 500

Cell Number		1	2*	3*	4	5	Avg.
Charge (OC = 18%)	m%	35	—	—	32	15	—
	V_f	2.12	—	—	2.12	2.10	—
Discharge	p%	25	—	—	25	25	—
	V_p	1.14	—	—	1.24	1.24	—
	V_e	1.06	—	—	1.20	1.20	—
Electrolyte Addition	Cum. Amt (cc)	7.00	—	—	5.00	5.00	—

Note: #2 and #3 failed at cycles 477 and 462, respectively.

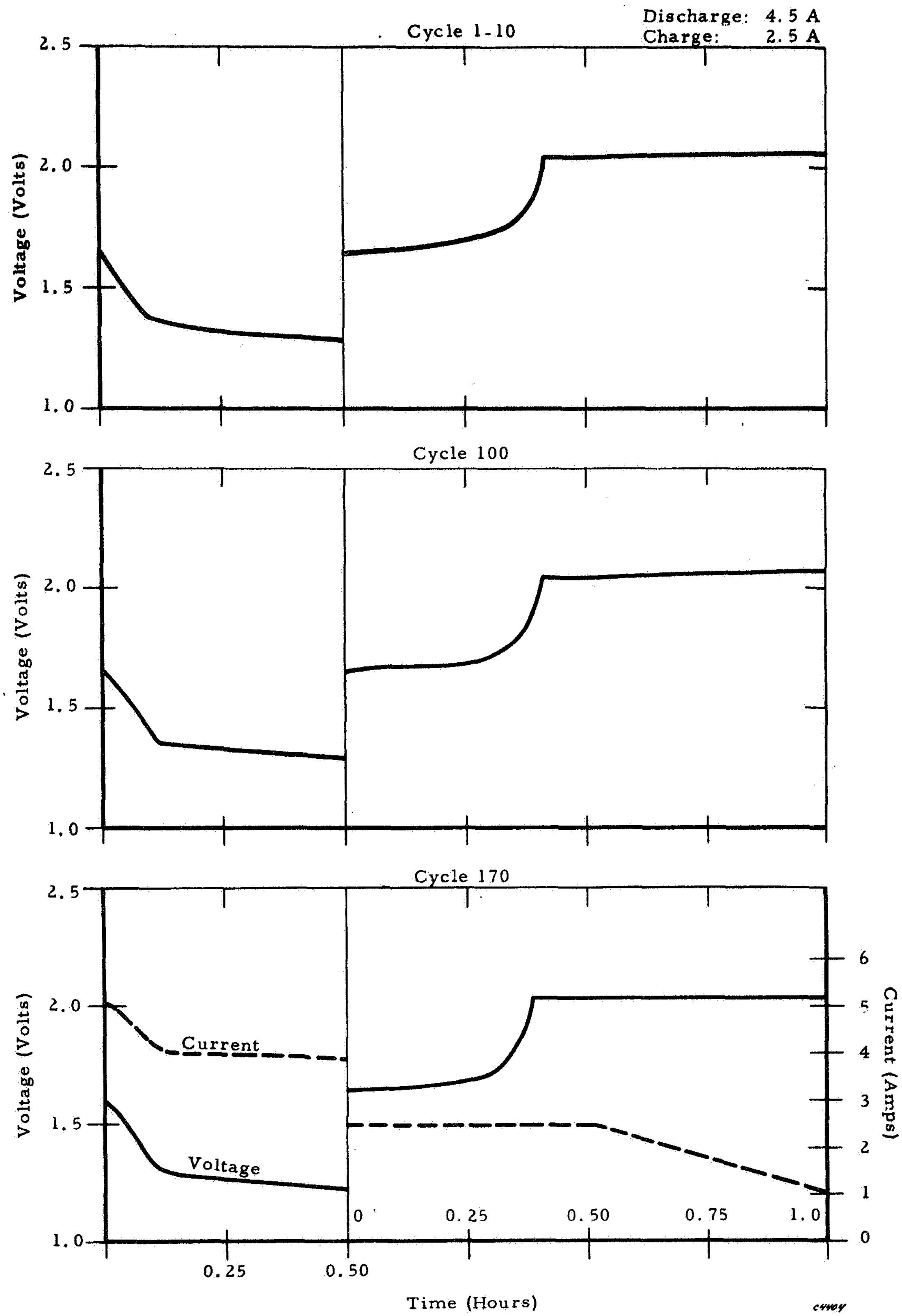


Figure 38. Group A (Environmental Test) Typical Cell

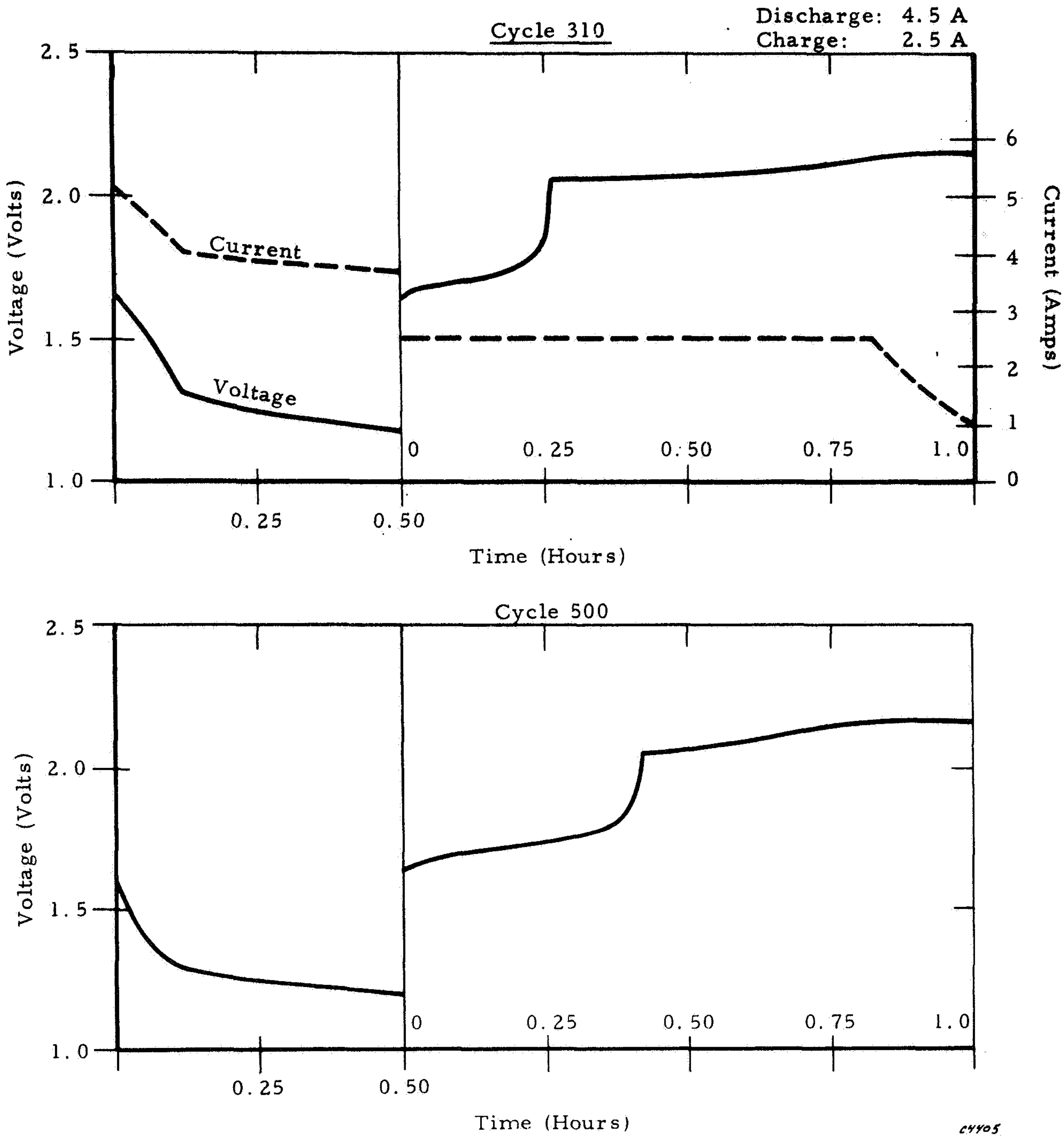


Figure 39. Group A (Environmental Test) Typical Cell

TABLE XLVI

UNIFORMITY STUDY, GROUP B (CONTROL)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.5 A for 1.0 hr
Voltage Limit: 2.05 V/cell
Temperature: 25°C

Cycle 1-10

Cell Number		6	7	8	9	10	Avg.
Charge	m%	44	44	44	44	44	44
(OC = -7%)	V_f	2.05	2.05	2.04	2.05	2.06	2.05
Discharge	p%	22	22	22	22	22	22
	V_p	1.31	1.33	1.32	1.32	1.31	1.32
	V_e	1.28	1.29	1.30	1.30	1.29	1.29
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

Cycle 100

Charge	m%	38	38	38	38	38	38
(OC = -3%)	V_f	2.08	2.09	2.06	2.09	2.09	2.08
Discharge	p%	19	19	19	19	19	19
	V_p	1.29	1.32	1.31	1.33	1.29	1.31
	V_e	1.24	1.27	1.28	1.30	1.25	1.27
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

TABLE XLVII

UNIFORMITY STUDY, GROUP B (CONTROL)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.5 A for 1.0 hr
Voltage Limit: 2.05 V/cell
Temperature: 25°C

Cycle 170

Cell Number		6	7	8	9	10	Avg.
Charge	m%	31	62	31	38	31	39
(OC = -2%)	V_f	2.09	1.97	2.05	2.01	2.10	2.04
Discharge	p%	25	25	25	25	25	25
	V_p	1.29	1.29	1.29	1.30	1.27	1.29
	V_e	1.26	1.14	1.26	1.26	1.24	1.23
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

Cycle 310*

Charge	m%	22	22	22	22	22	22
(OC = +25%)	V_f	2.14	2.12	2.15	2.17	2.13	2.14
Discharge	p%	31	31	31	31	31	31
	V_p	1.25	1.29	1.26	1.25	1.28	1.27
	V_e	1.21	1.26	1.23	1.21	1.25	1.23
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

*After 200 cycles, voltage limit raised to 2.15 V/cell, and current to 2.75A.

TABLE XLVIII

UNIFORMITY STUDY, GROUP B (CONTROL)

Regime: Discharge: 4.5 A for 0.5 hr

Charge: A for 1.0 hr

Voltage Limit: 2.15 V/cell

Temperature: 25°C

Cycle 500

Cell Number		6	7	8	9	10	Avg.
Charge	m%	20	20	20	20	20	20
(OC = 20%)	V_f	2.18	2.12	2.14	2.20	2.18	2.16
Discharge	p%	30	30	30	30	25	29
	V_p	1.32	1.32	1.32	1.15	1.34	1.29
	V_e	1.28	1.28	1.28	1.05	1.30	1.28
Electrolyte Addition	Cum. Amt (cc)	4.0	5.0	9.0	9.5	3.0	6.0

Cycle 700

Charge	m%	17	15	15	17	15	16
(OC = 15%)	V_f	2.20	2.09	2.19	2.10	2.18	2.15
Discharge	p%	30	30	30	30	30	30
	V_p	1.24	1.22	1.32	1.20	1.30	1.26
	V_e	1.16	1.16	1.26	1.04	1.28	1.18
Electrolyte Addition	Cum. Amt (cc)	11.5	12.0	16.0	16.5	13.0	13.8

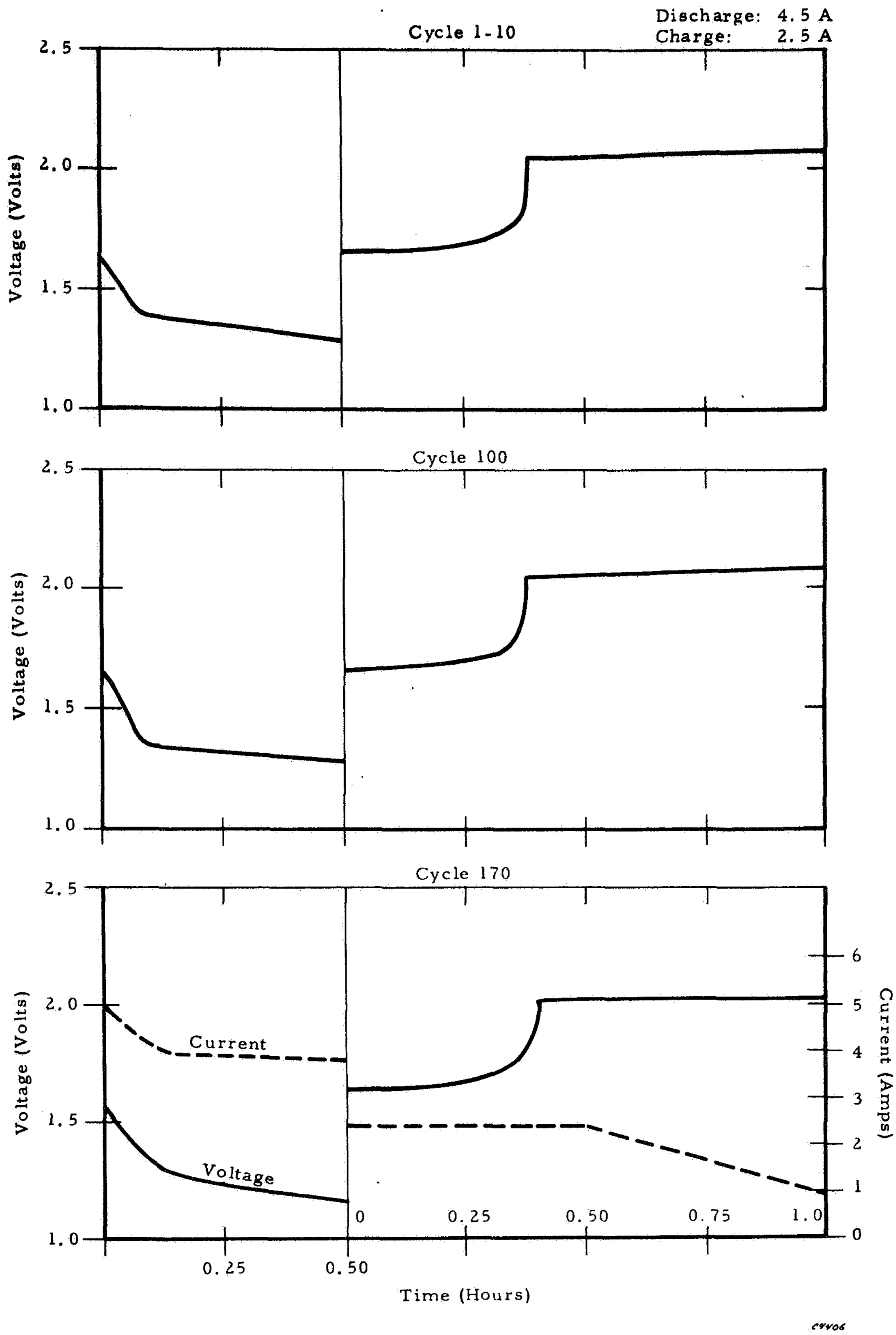


Figure 40. Group B (Control) Typical Cell

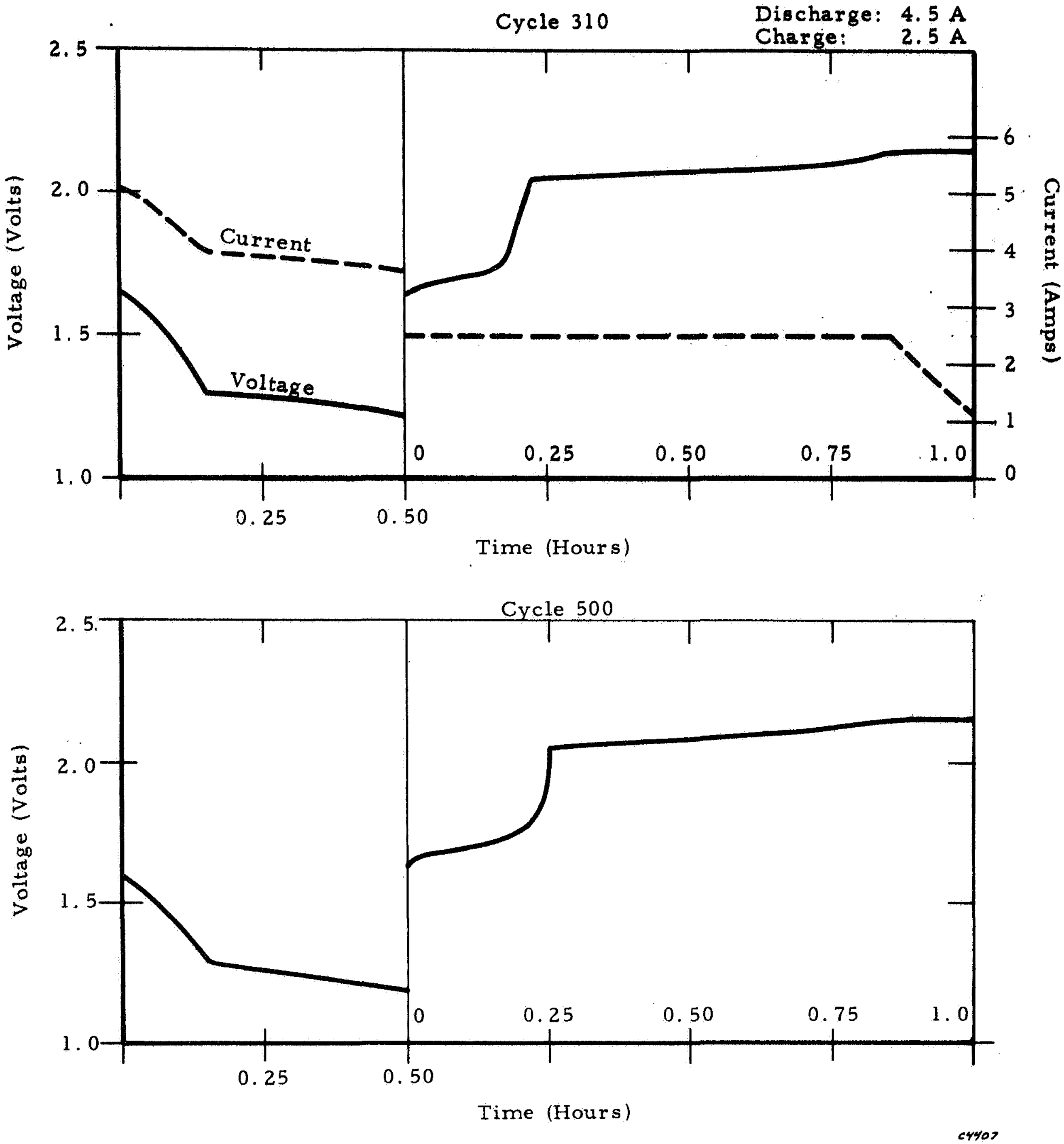


Figure 41. Group B (Control) Typical Cell

TABLE IL

UNIFORMITY STUDY, GROUP C (PRESSURE GAUGE)

Regime: Discharge: 4.5 A for 0.5 hr

Charge: 2.5 A for 1.0 hr

Voltage Limit: 2.05 V/cell

Temperature: 25°C

Cycle 1-10

Cell Number		11	12	13	14	15	Avg.
Charge	m%	50	50	50	50	50	50
(OC = -4%)	V_f	2.06	2.06	2.06	2.06	2.06	2.06
Discharge	p%	16	16	16	16	16	16
	V_p	1.27	1.26	1.26	1.26	1.26	1.26
	V_e	1.24	1.23	1.23	1.23	1.23	1.23
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0
Pressure	psig	6.0	6.0	5.0	5.0	6.0	5.6

Cycle 100

Charge	m%	42	42	42	42	42	42
(OC = -1%)	V_f	2.09	2.08	2.08	2.08	2.07	2.08
Discharge	p%	19	19	19	19	19	19
	V_p	1.28	1.26	1.28	1.28	1.28	1.28
	V_e	1.23	1.21	1.23	1.23	1.23	1.23
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0
Pressure	psig	33	22	30	29	21	27

TABLE L

UNIFORMITY STUDY, GROUP C (PRESSURE GAUGE)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.5 A for 1.0 hr
Voltage Limit: 2.05 V/cell
Temperature: 25°C

Cycle 170

Cell Number		11	12	13	14	15	Avg.
Charge	m%	31	31	31	31	34	31
(OC = +1%)	V_f	2.04	2.05	2.08	2.02	2.03	2.04
Discharge	p%	25	25	25	25	25	25
	V_p	1.22	1.18	1.19	1.18	1.23	1.20
	V_e	1.20	1.13	1.15	1.14	1.19	1.16
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0
Pressure	psig	33	26	29	30	24	28

Cycle 310*

Charge	m%	26	26	26	26	26	26
(OC = +16%)	V_f	2.16	2.16	2.09	2.15	2.10	2.13
Discharge	p%	25	25	25	25	25	25
	V_p	1.24	1.19	1.23	1.22	1.24	1.22
	V_e	1.17	1.12	1.20	1.17	1.20	1.17
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0
Pressure	psig	30	28	30	31	25	29

*After 200 cycles, voltage limit raised to 2.13 V/cell, and current to 2.75 A.

TABLE LI

UNIFORMITY STUDY, GROUP C (PRESSURE GAUGE)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.75 A for 1.0 hr
Voltage Limit: 2.15V/cell
Temperature: 25°C

Cycle 500

Cell Number		11	12	13	14	15	Avg.
Charge	m%	15	18	27	15	15	18
(OC = 17%)	V_f	2.12	2.20	2.12	2.16	2.16	2.15
Discharge	p%	30	30	30	30	30	30
	V_p	1.18	1.30	1.24	1.24	1.30	1.25
	V_e	1.12	1.20	1.16	1.16	1.26	1.18
Electrolyte Addition	Cum. Amt (cc)	4.0	6.0	4.0	5.0	5.0	5.0
Pressure	psig	29	28	30	28	26	28

Cycle 700

Charge	m%	17	20	20	24	20	20
(OC =	V_f	2.24	2.26	2.12	2.13	2.24	2.20
Discharge	p%	30	30	30	25	30	30
	V_p	1.20	1.20	1.18	1.20	1.22	1.20
	V_e	1.15	1.10	1.00	1.04	1.18	1.09
Electrolyte Addition	Cum. Amt (cc)	8.0	11.0	9.0	10.0	11.0	10.0
Pressure	psig	32	30	29	28	25	29

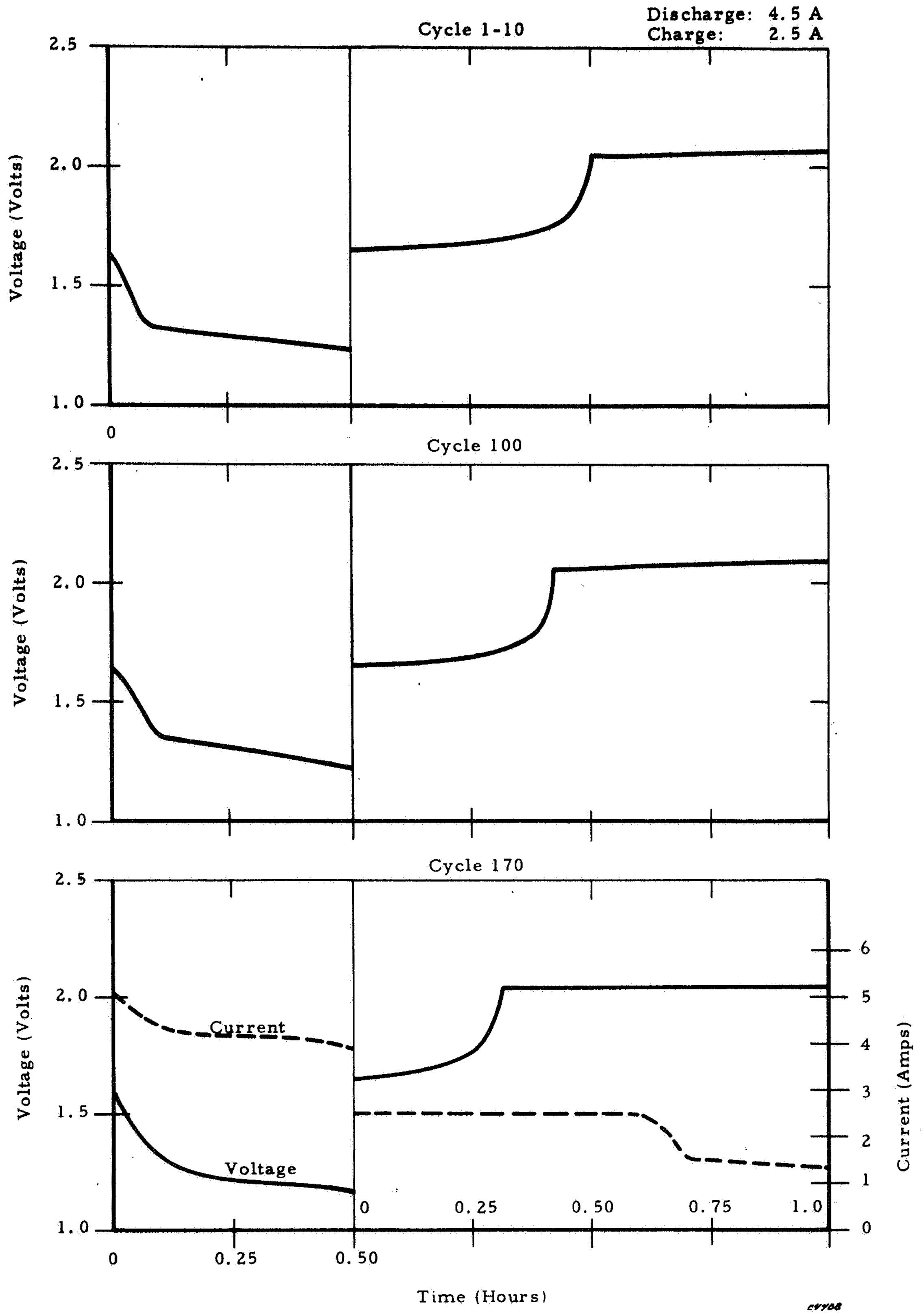


Figure 42. Group C (Pressure Gauge) Typical Cell

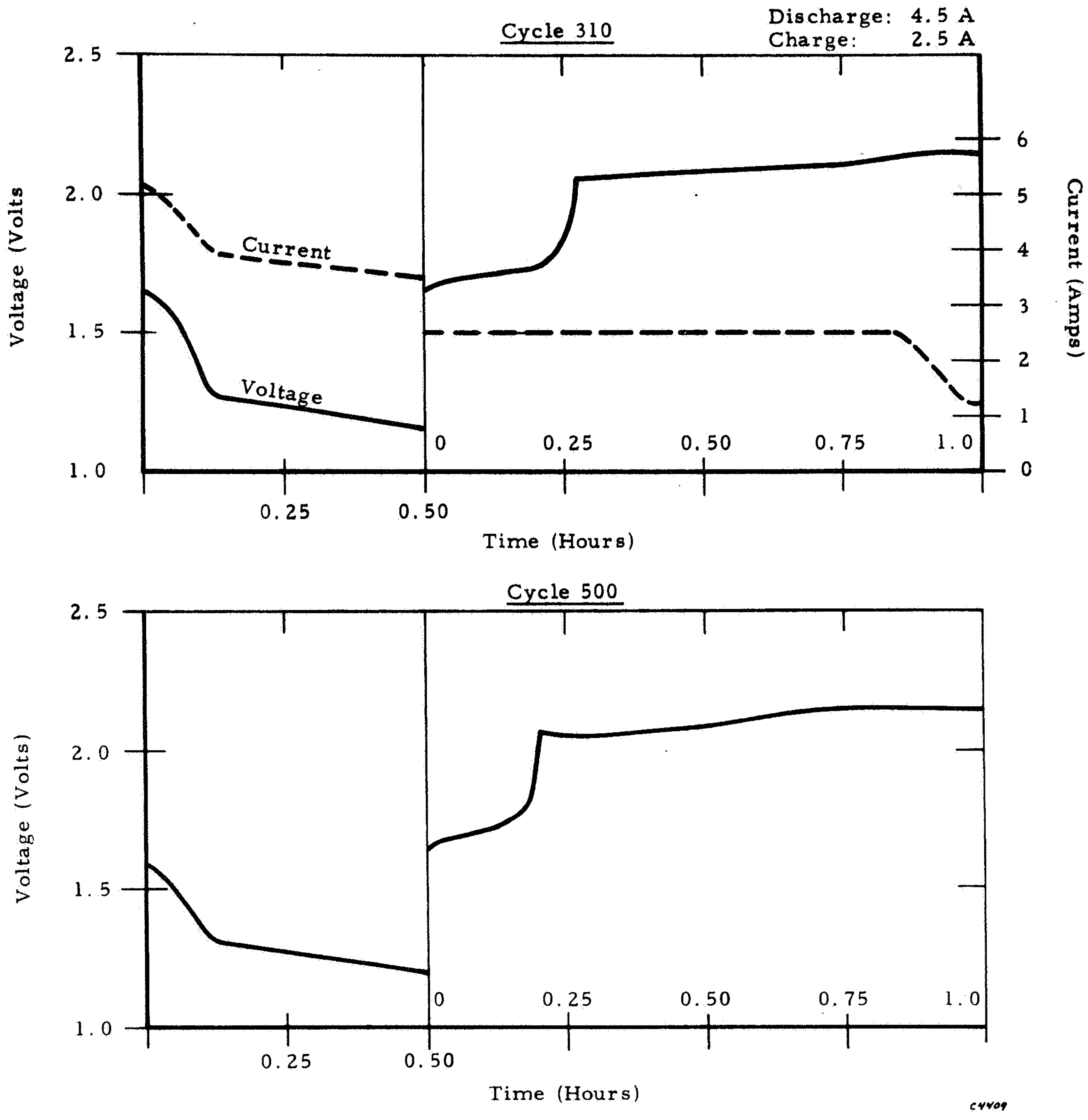


Figure 43. Group C (Pressure Gauge) Typical Cell

TABLE LII

UNIFORMITY STUDY, GROUP D (REPEAT)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.5 A for 1.0 hr
Voltage Limit: 2.15 V/cell
Temperature: 25°C

Cycle 1-10

Cell Number		16*	22	23	24	25	Avg.
Charge	m%	43	43	43	43	43	43
(OC = 0%)	V_f	2.16	2.13	2.10	2.11	2.11	2.12
Discharge	p%	20	20	20	20	20	20
	V_p	1.23	1.26	1.29	1.28	1.27	1.27
	V_e	1.19	1.23	1.27	1.25	1.25	1.24
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0
Pressure	psig	18	—	—	—	—	—

Cycle 100

Charge	m%	33	33	33	33	33	33
(OC = 5%)	V_f	2.14	2.12	2.10	2.10	2.11	2.11
Discharge	p%	25	25	25	25	25	25
	V_p	1.18	1.27	1.32	1.30	1.30	1.27
	V_e	1.14	1.23	1.28	1.26	1.26	1.23
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0
Pressure	psig	29	—	—	—	—	—

*with pressure gauge.

TABLE LIII

UNIFORMITY STUDY, GROUP D (REPEAT)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.5 A for 1.0 hr
Voltage Limit: 2.15 V/cell
Temperature: 25°C

Cycle 200

Cell Number		16*	22	23	24	25	Avg.
Charge	m%	18	18	18	18	18	18
(OC = 8%)	V_f	2.20	2.15	2.16	2.06	2.21	2.16
Discharge	p%	33	33	33	33	33	33
	V_p	1.22	1.25	1.25	1.26	1.31	1.26
	V_e	1.18	1.22	1.21	1.22	1.27	1.22
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0
Pressure	psig	34	—	—	—	—	—

Cycle 300

Charge	m%	25	25	25	25	25	25
(OC = 7%)	V_f	2.16	2.16	2.16	2.16	2.04	2.14
Discharge	p%	30	25	25	25	20	25
	V_p	1.20	1.20	1.28	1.32	1.24	1.25
	V_e	1.16	1.18	1.24	1.28	1.10	1.19
Electrolyte Addition	Cum. Amt (cc)	2	2	2	2	2	2
Pressure	psig	25	—	—	—	—	—

* with pressure gauge.

TABLE LIV

UNIFORMITY STUDY, GROUP D
(REPEAT)

Regime: Discharge: 4.5 A for 0.5 hr
Charge: 2.5 A for 1.0 hr
Voltage Limit: 2.15 V/cell
Temperature: 25°C

Cycle 450

Cell Number		16*	17	18	19	20	Avg.
Charge	m%	30	20	25	30	25	26
(OC =	V_f	2.10	2.16	2.15	2.12	2.12	2.18
Discharge	p%	20	30	25	25	25	25
	V_p	1.22	1.22	1.22	1.24	1.26	1.24
	V_e	1.17	1.19	1.19	1.16	1.20	1.18
Electrolyte Addition	Cum. Amt (cc)	6	8	8	9	8	8
Pressure	psig	27	--	--	--	--	--

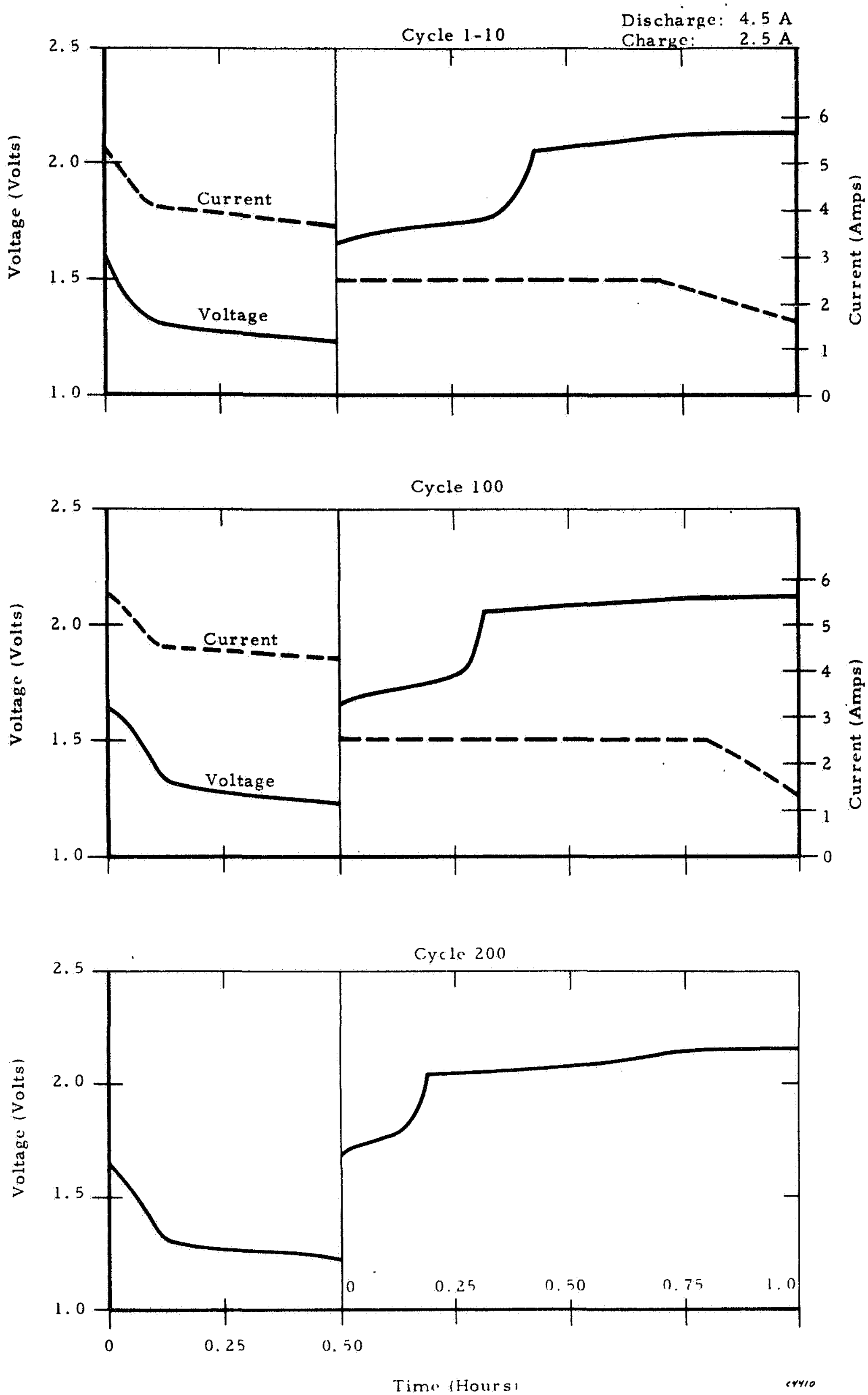


Figure 44. Group D: Repeat of Controls With Higher Voltage Limit

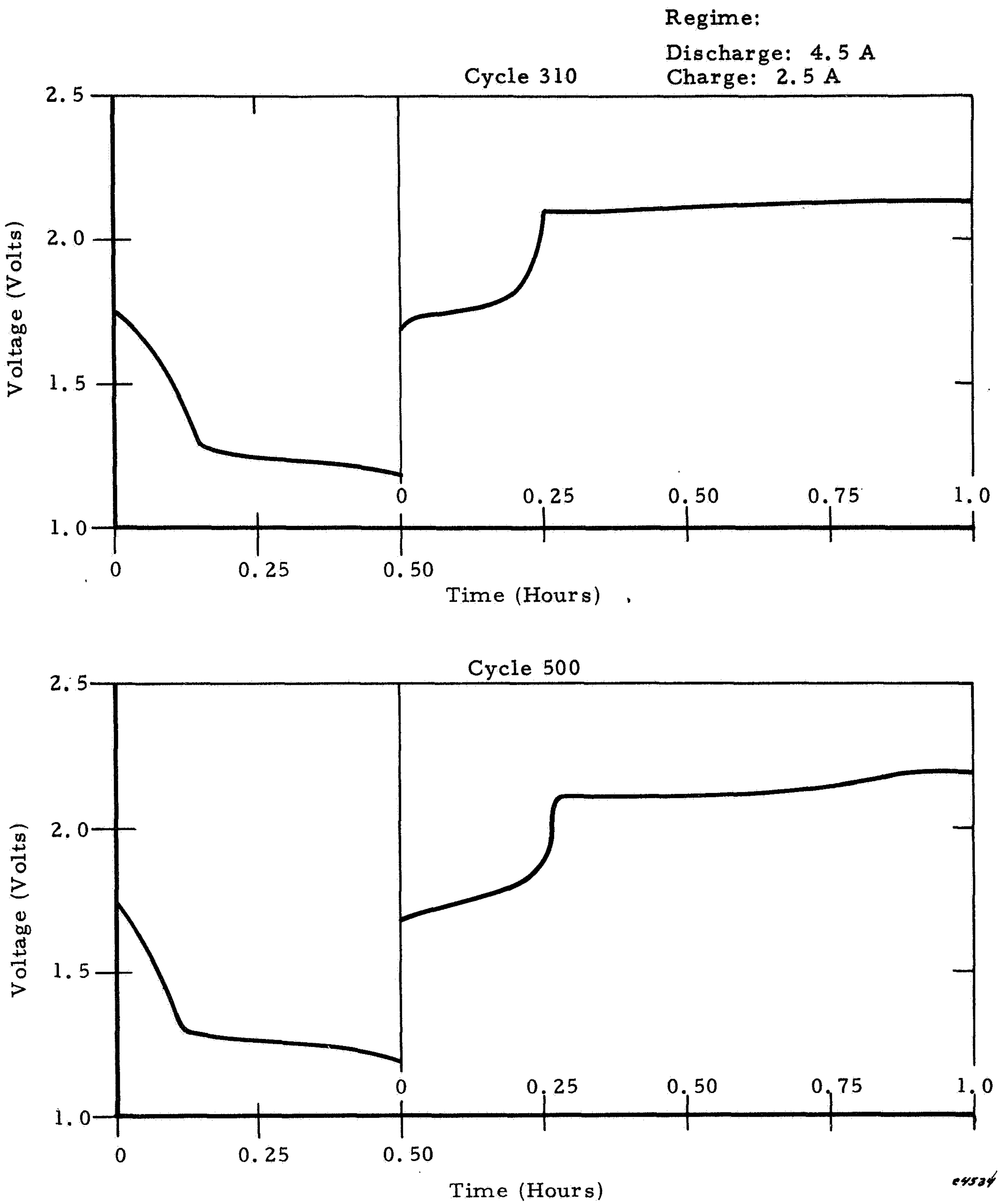


Figure 45. Group D: Repeat of Controls With Higher Voltage Limit

TABLE LV

GROUP ZL-40 — TEST SUMMARY

Group	Description	Cell No.	Total Cycles	Final Capacity (Ah)
A	Environmentally Tested	1	557	2.9 Ah
		2	477	2.4
		3	462	2.5
		4	590	3.2
		5	527	3.1
		Average	523	2.8 Ah
B	Controls	6	961	0.1 Ah
		7	960	2.6
		8	760	0.8
		9	730	2.0
		10	843	2.0
		Average	851	1.5 Ah
C	Pressure Gauges	11	789	2.7 Ah
		12	729	3.6
		13	880	2.6
		14	729	2.5
		15	866	2.6
		Average	787	2.8 Ah
D	Repeat of Controls	16*	531	1.6 Ah
		17	550	1.3
		18	464	1.2
		19	464	1.2
		20	457	2.4
		Average	487	1.5 Ah
		Grand Average	662	

*with pressure gauge

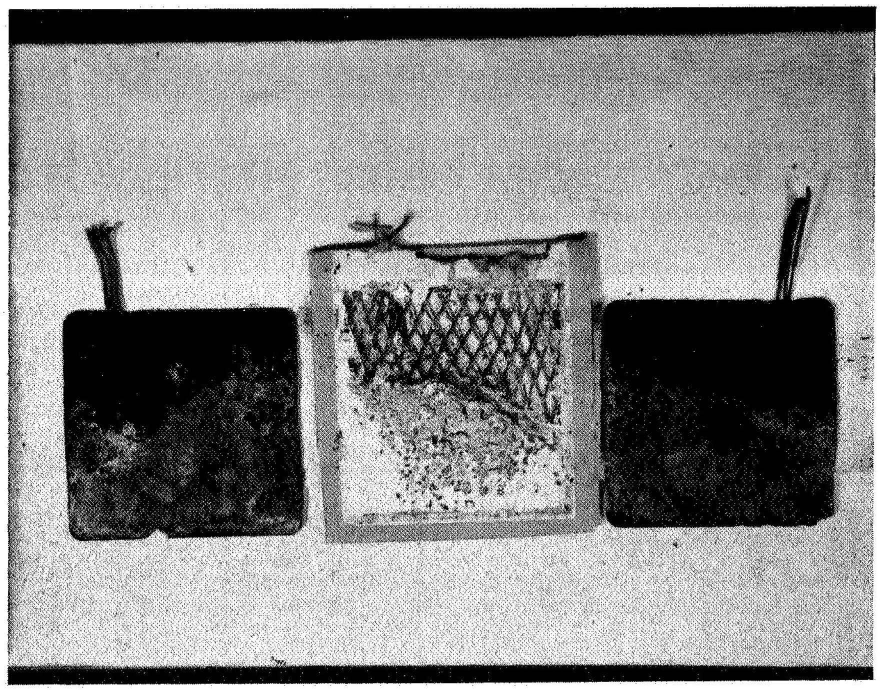


Figure 46. ZL-40-1 Cell: Condition of Electrodes After 557 Cycles at 45% Depth of Discharge of Rated Capacity

C4521

Figure 47. Silver-Zinc Cells of Final Design for Task III

Twenty-five cells from each lot were delivered to the Naval Ammunition Depot, at Crane, Indiana, Battery Testing Laboratory, for tests as directed by NASA.

Ten cells were retained in our laboratory for various tests as summarized in Table LVI.

For the sake of convenience and easy reference, each set of test conditions is coded A through F. Each lot is numbered 1 through 6. Tests A, B, and C apply to odd-numbered lots (1, 3, 5) and tests D, E, and F apply to even-numbered lots (2, 4, 6)

The remainder of the program covers specifically such tests and their correlation. Three independent variables are being considered:

- Current density or current, I
- Temperature, T
- Cycling period or depth of discharge, D

While two are maintained constants, the other variable is varied on 2 or 3 levels.

An overall view of the interrelationship of variables and test groups to be compared is given in Table LVII.

Each cell has a bi-numeral code: the first number refers to the lot (1 through 6) and the second number refers to the cell (1 through 25). For example, 1-24 means Cell #24 of Lot #1. The complete cycling data are presented in Table LVIII and Table LIX. Ranges and averages of cycles extracted from these tables are given in Table LX.

Each cycling test condition for each lot is covered by tables and curves giving pertinent data at various cycles.

Lot #1 —	Test A	Tables LXI-LXIII	Figures 48-50
	Test B	Tables LXIV-LXV	
	Test C	Tables LXVI-LXVIII	Figure 51

TABLE LVI

TASK III

CELL TEST DISTRIBUTION

	Test Groups					
Test Conditions	A	B	C	D	E	F
Orbit Period	1.5 hr	1.5 hr	24 hrs	2 hrs	2 hrs	Stand
Discharge/Charge	0.5/1 hr	0.5/1 hr	1.2/22.8 hr	0.58/1.42 hr	0.58/1.42 hr	
Current	3 A	3 A	3 A	3 A	5.4 A	
Temperature	25°C	100°C	25°C	25°C	25°C	25°C
Number of Cells per Lot	4	4	2	4	4	2
Lot Numbers	#1, #3, #5			#2, #4, #6		

TABLE LVII

TASK III

CORRELATION OF VARIABLES

<table>
<tr><th rowspan="3">Independent Constants</th><th rowspan="3">Related Variables</th><th colspan="4">Levels</th><th rowspan="3">Test Groups</th></tr>
<tr><th rowspan="2">Period Time</th><th rowspan="2">Discharge Time</th><th colspan="2">Depth (%)</th></tr>
<tr><th>of actual (7.5 Ah)</th><th>of rated (5 Ah)</th></tr>
<tr><td>Current: 3 A
Temperature: 25°C</td><td>Period
Depth</td><td>1.5 hr
2 hrs
24 hrs</td><td>0.5 hr
0.582 hr
1.2 hrs</td><td>20%
23%
48%</td><td>30%
35%
72%</td><td>A
D
C</td></tr>
<tr><td rowspan="3">Temperature: 25°C
Period: 2 hrs</td><td rowspan="3">Current Density
Current
Depth</td><td rowspan="2">Current Density</td><td rowspan="2">Current</td><td colspan="2">Depth (%)</td><td rowspan="2"></td></tr>
<tr><td>of actual (7.5 Ah)</td><td>of rated (5 Ah)</td></tr>
<tr><td>25 mA/cm^2
45 mA/cm^2</td><td>3 A
5.4 A</td><td>23%
42%</td><td>35%
63%</td><td>D
E</td></tr>
<tr><td rowspan="2">Period and Depth
(1.5 hr; 20%)
Current: 3 A</td><td rowspan="2">Temperature</td><td rowspan="2"></td><td rowspan="2"></td><td colspan="2">Temperature</td><td rowspan="2">A
B</td></tr>
<tr><td colspan="2">25°C
100°C</td></tr>
</table>

TABLE LVIII

CYCLING DATA OF CELLS OF LOTS 1, 3, 5

Test / Lot No.	A		B		C	
	Cell No.	Cycle	Cell No.	Cycle	Cell No.	Cycle
1	1-26	2002	1-30	328	1-34	51
	1-27	895*	1-31	651	1-35	159
	1-28	1493	1-32	330		
	1-29	997*	1-33	325		
3	3-26	1388	3-30	456	3-34	74
	3-27	1543	3-31	225	3-35	100
	3-28	1570	3-32	460		
	3-29	843*	3-33	112*		
	3-36	960*	3-29	405		
			3-40	426		
5	5-26	1025	5-30	291	5-34	64
	5-27	1356	5-31	409	5-35	101
	5-28	1604	5-32	290		
	5-29	1401	5-33	648		

*Technical failure: Cell was capable of cycling, but capacity was less than 4 Ah and test was discontinued for examination of cell as required by the work statement (cells not counted in average).

TABLE LIX

DATA ON CELLS OF LOTS 2, 4, 6

Lot No. \ Test	D		E			F			
	Cell No.	Cycle		Cell No.	Cycle	Cell No.	Days	Residual Capacity	Capacity after Rechg
2	2-26	493*	I	2-30	56+	2-34	178	7.0 Ah	7.0 Ah
	2-27	453*		2-31	55+	2-35	178	7.1 Ah	7.2 Ah
	2-28	1783		2-32	126				
	2-29	1233		2-33	124				
			II	12-21	158				
				12-22	405				
				12-23	461				
				12-24	364				
			III	2-36	245				
				2-37	225				
				2-38	352				
4	4-26	1117		4-39	276	4-34	177	4.9 Ah	6.3 Ah
	4-27	1177		4-31	351	4-35	177	5.9 Ah	6.7 Ah
	4-28	1072		4-32	229				
	4-29	1218		4-33	164				
6	6-26	983		6-30	157	6-34	186	5.8 Ah	7.3 Ah
	6-27	1028		6-31	156	6-35	186	6.2 Ah	7.0 Ah
	6-28	1298		6-32	167				
	6-29	1570		6-33	77				

Note: In Lot No. 2, Test E, Group I = sealed, 40% KOH;
Group II = vented, 30% KOH, Group III = vented, 40% KOH.

*Technical failure: Cell was capable of cycling, but capacity was less than 4 Ah and test was discontinued for examination of cell as required by the work statement (cell not counted in average).

+Cells had 4.75 Ah and 5.75 Ah respectively, but could not cycle any longer in a sealed state. They were discontinued.

TABLE LX

RANGE (AVERAGE) OF CYCLING DATA

Lot \ Test	A	B	C
1	1000 - 2002 (1747)	325 - 651 (410)	51 - 159 (105)
3	1388 - 1570 (1500)	225 - 460 (394)	74 - 100 (87)
5	1025 - 1604 (1346)	290 - 648 (410)	64 - 101 (83)
Grand Average	1487	403	92
Lot \ Test	**D**	**E**	**F***
2	1233 - 1783 (1508)	55 - 461 (233)	7.0 Ah
4	1072 - 1218 (1146)	164 - 351 (255)	5.4 Ah
6	983 - 1570 (1220)	77 - 167 (139)	6.0 Ah
Grand Average	1291	209	6.1 Ah

*Residual capacity after 6 month charged stand.

TABLE LXI

UNIFORMITY STUDY, LOT #1, TEST A

Regime: Discharge: 3.0 A for 0.5 hr
Charge: 1.7 A for 1.0 hr
Voltage Limit: 2.13 V/cell
Temperature: 25°C

Cycle 10

Cell Number		26	27	28	29		Avg.
Charge	m%	55	55	55	55		55
(OC = 4%)	V_f	2.04	2.04	2.04	2.03		2.04
Discharge	p%	11	11	11	11		11
	V_p	1.32	1.33	1.32	1.33		1.33
	V_e	1.27	1.28	1.27	1.28		1.28
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

Cycle 300

Charge	m%	26	26	26	26		26
(OC = 4.3)	V_f	2.12	2.12	2.11	2.15		2.13
Discharge	p%	25	25	25	25		25
	V_p	1.37	1.34	1.30	1.36		1.34
	V_e	1.28	1.26	1.24	1.29		1.27
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

TABLE LXII

UNIFORMITY STUDY, LOT #1, TEST A

Regime: Discharge: 3.0 A A for 0.5 hr
Charge: 1.70 A A for 1.0 hr
Voltage Limit: 2.13 V/cell
Temperature: 25 °C

Cycle 600

Cell Number		26	27	28	29		Avg.
Charge	m%	22	22	22	22		22
(OC = 17 %)	V_f	2.15	2.12	2.12	2.11		2.13
Discharge	p%	28	28	28	28		28
	V_p	1.31	1.28	1.29	1.28		1.29
	V_e	1.24	1.21	1.23	1.22		1.23
Electrolyte Addition	Cum. Amt (cc)	2.0	0	0	1.5		1.75

Cycle 900

Charge	m%	22	*	22	22		22
(OC = 17 %)	V_f	2.07		2.17	2.13		2.12
Discharge	p%	29		29	29		29
	V_p	1.27		1.29	1.36		1.31
	V_e	1.18		1.15	1.30		1.21
Electrolyte Addition	Cum. Amt (cc)	9.5	5	8.5	5.0		6.2

*Note: Cell #27 failed after 895 cycles.

TABLE LXIII

UNIFORMITY STUDY, LOT #1, TEST A

Regime: Discharge: 3.0 A for 0.5 hr

Charge: 1.70 A for 1.0 hr

Voltage Limit: 2.13 V/cell

Temperature: 25 °C

Cycle 1200

Cell Number		26		28	29		Avg.
Charge	m%	17		17	*		17
(OC = 4 %)	V_f	2.12		2.14			2.13
Discharge	p%	32		32			32
	V_p	1.24		1.28			1.26
	V_e	1.14		1.16			1.15
Electrolyte Addition	Cum. Amt (cc)	9.5		8.5			8

*Note: Cell #29 failed after 997 cycles.

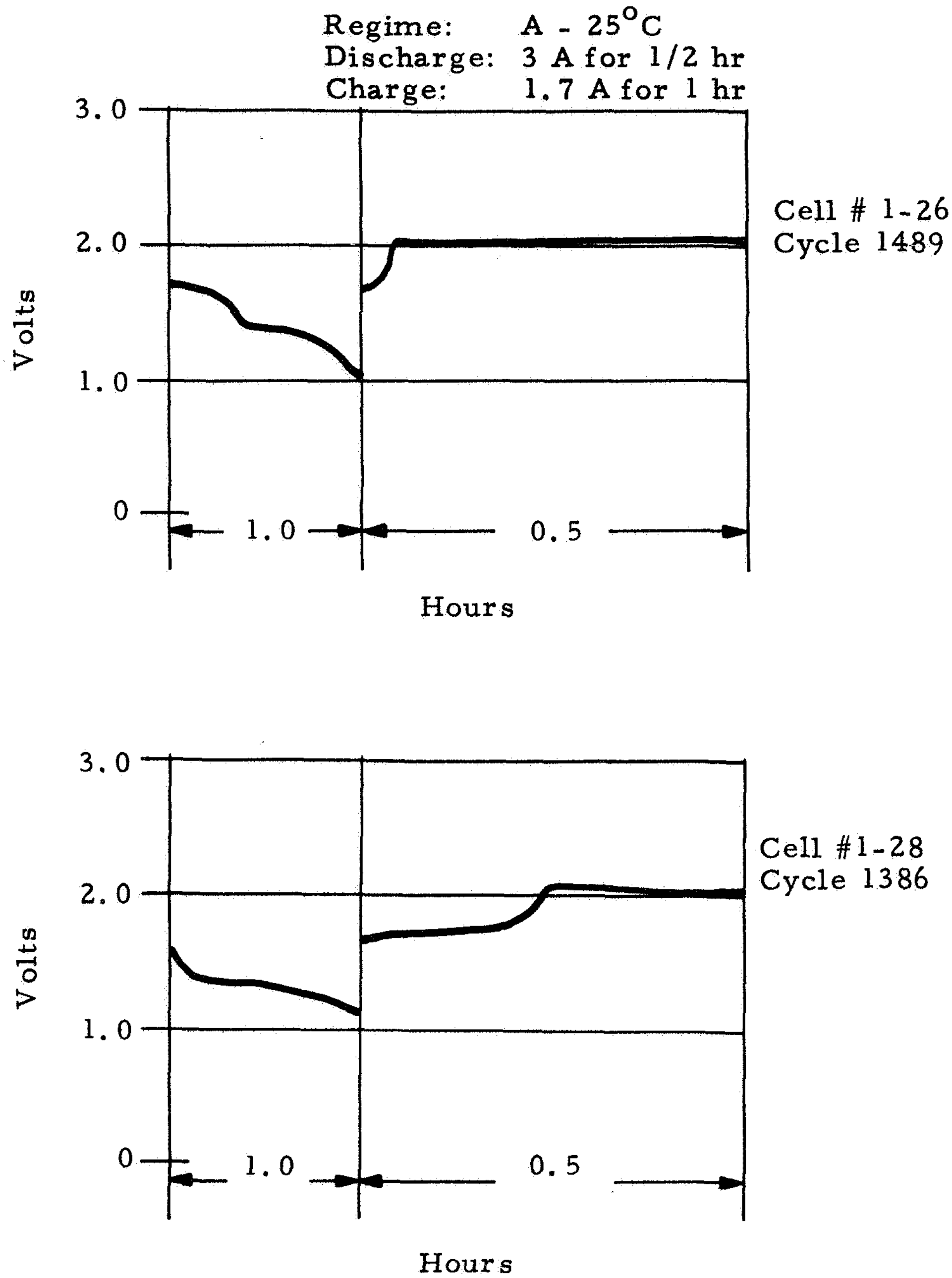


Figure 48. Cycling Curves, Lot #1 - Test A

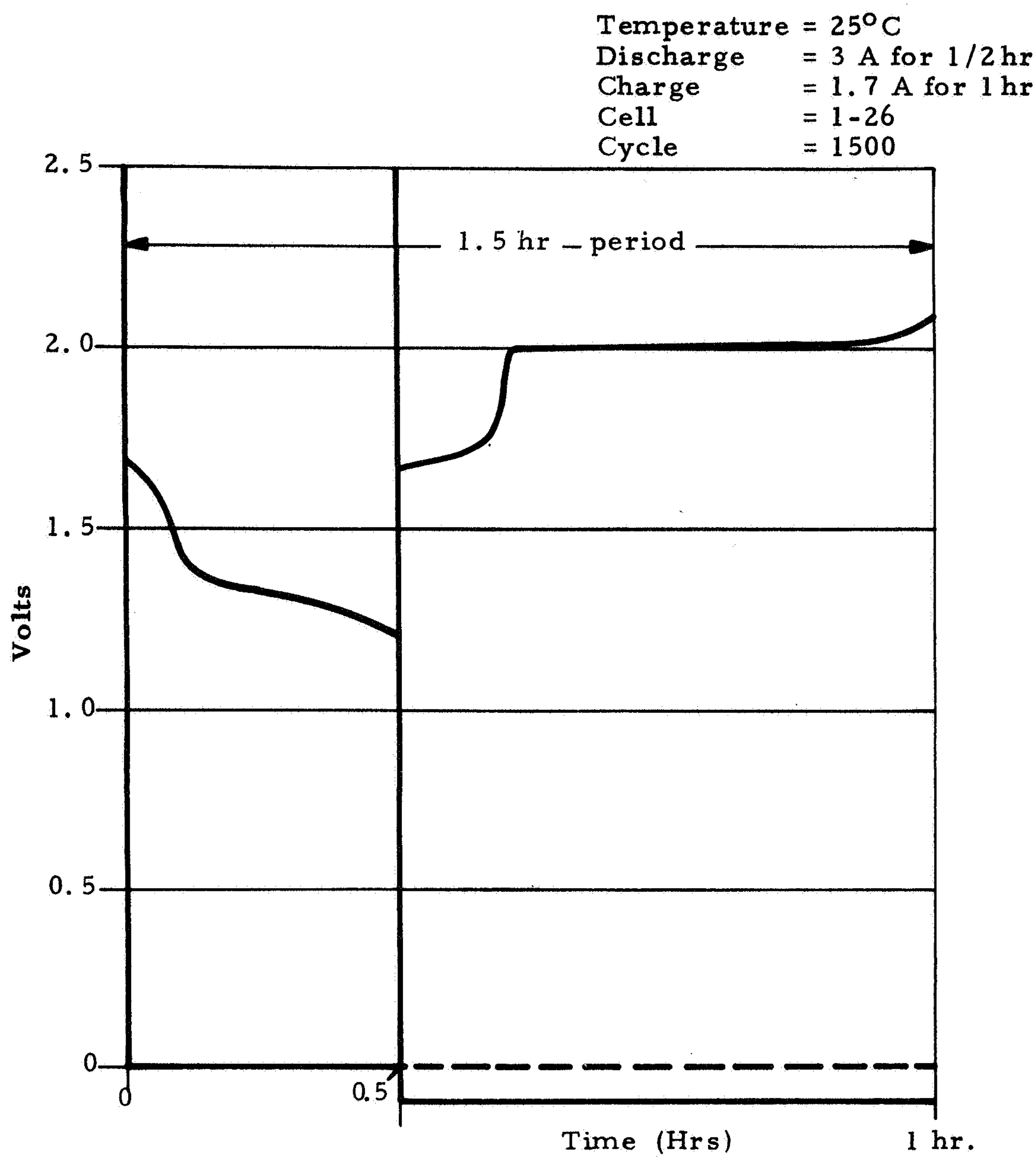


Figure 49. Cycling Curves, Lot #1 - Test A

Temperature: $25^{\circ}C$
Discharge: 3 A for 0.5 hr
Charge: 1.7 A for 1 hr
Cell: 1-26
Cycle: 1800

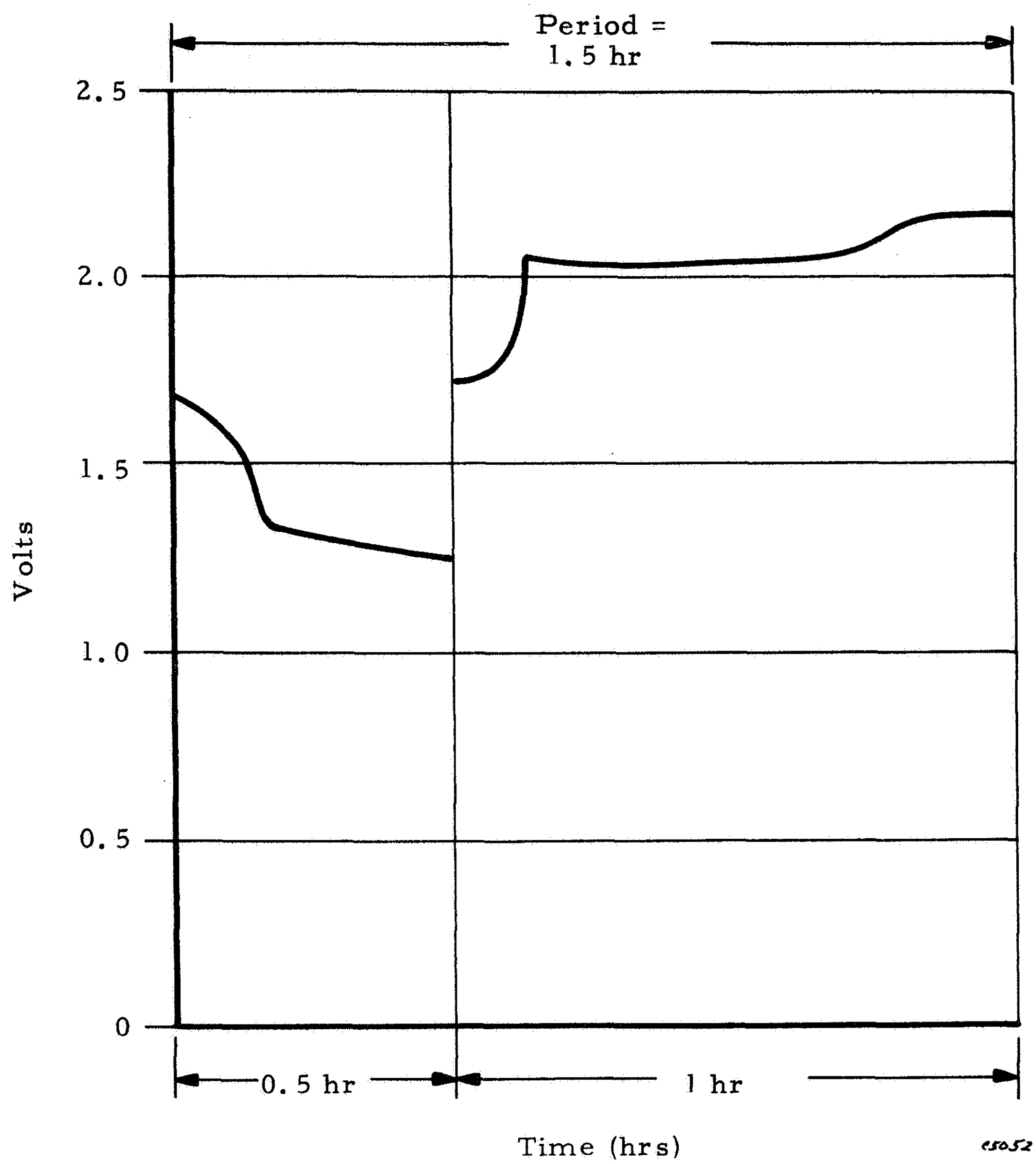


Figure 50. Cycling Curves for Lot 1 - Test A

TABLE LXIV

UNIFORMITY STUDY, LOT #1, TEST B

Regime: Discharge: 3.0 A for 0.5 hr
Charge: 1.6 A for 1.0 hr
Voltage Limit: 2.02 V/cell
Temperature: 100°C

Cycle 1-17

Cell Number		30	31	32	33		Avg.
Charge	m%	49	49	49	49		49
(OC = 2%)	V_f	2.00	1.99	1.99	1.99		1.99
Discharge	p%	14	14	14	14		14
	V_p	1.45	1.46	1.45	1.45		1.45
	V_e	1.45	1.46	1.45	1.45		1.45
Electrolyte Addition	Cum. Amt (cc)	2.0	0	0	0		0.5

Cycle 100

Charge	m%	35	35	35	35		35
(OC = 3.4%)	V_f	2.03	1.99	1.99	1.99		2.00
Discharge	p%	28	28	28	28		28
	V_p	1.45	1.45	1.44	1.45		1.45
	V_e	1.44	1.45	1.43	1.44		1.44
Electrolyte Addition	Cum. Amt (cc)	13.0	11.0	11.0	5.0		10.0

TABLE LXV

UNIFORMITY STUDY, LOT #1, TEST B

Regime: Discharge: 3.0 A for 0.5 hr
Charge: 1.6 A for 1.0 hr
Voltage Limit: 2.02 V/cell
Temperature: 25°C

Cycle 200

Cell Number		30	31	32	33		Avg.
Charge	m%	41	38	38	31		40
(OC = 6.5%)	V_f	2.03	2.00	2.02	2.01		2.02
Discharge	p%	19	19	19	19		19
	V_p	1.41	1.43	1.41	1.42		1.42
	V_e	1.38	1.42	1.40	1.41		1.40
Electrolyte Addition	Cum. Amt (cc)	20.5	19.0	20.0	13.0		18.0

Cycle 300

Charge	m%	40	26	40	47		38
(OC = 14%)	V_f	2.02	2.04	2.00	2.00		2.02
Discharge	p%	14	25	14	14		17
	V_p	1.40	1.41	1.41	1.38		1.40
	V_e	1.32	1.40	1.38	1.34		1.36
Electrolyte Addition	Cum. Amt (cc)	33.5	33.0	34.0	27.5		32.0

TABLE LXVI

UNIFORMITY STUDY, LOT #1, TEST C

Regime: Discharge: 3.0 A for 1.2 hr
Charge: 0.170 A for 22.8 hr
Voltage Limit: 2.04 V/cell
Temperature: 25°C

Cycle 1

Cell Number		34	35				Avg.
Charge	m%	32	32				32
(OC = 1%)	V_f	2.04	2.04				2.04
Discharge	p%	12	12				12
	V_p	1.36	1.36				1.36
	V_e	1.36	1.36				1.36
Electrolyte Addition	Cum. Amt (cc)	0	0				0

Cycle 30

Charge	m%	47	47				47
(OC = 3.9%)	V_f	2.04	2.04				2.04
Discharge	p%	4	4				4
	V_p	1.32	1.32				1.32
	V_e	1.27	1.27				1.27
Electrolyte Addition	Cum. Amt (cc)	0	0				0

TABLE LXVII

UNIFORMITY STUDY, LOT #1, TEST C

Regime: Discharge: 3.0 A for 0.5 hr
Charge: 0.170 A for 22.8 hr
Voltage Limit: ·2.04 V/cell
Temperature: 25 °C

Cycle 60

Cell Number		34*	35				Avg.
Charge	m%		43				
(OC = 0.5 %)	V_f		2.04				
Discharge	p%		4				
	V_p		1.36				
	V_e		1.26				
Electrolyte Addition	Cum. Amt (cc)	0	0				

*Note: Cell #34 failed after 51 cycles

Cycle 90

Charge	m%		39				
(OC = 2 %)	V_f		2.04				
Discharge	p%		4				
	V_p		1.36				
	V_e		1.26				
Electrolyte Addition	Cum. Amt (cc)		0				

TABLE LXVIII

UNIFORMITY STUDY, LOT #1, TEST C

Regime: Discharge: 3.0 A for 1.2 hr

Charge: 0.170 A for 22.8 hr

Voltage Limit: 2.04 V/cell

Temperature: 25 °C

Cycle 120

Cell Number			35				Avg.
Charge	m%		33				
(OC = 16.9 %)	V_f		2.04				
Discharge	p%		5.57				
	V_p		1.36				
	V_e		1.22				
Electrolyte Addition	Cum. Amt (cc)		0				

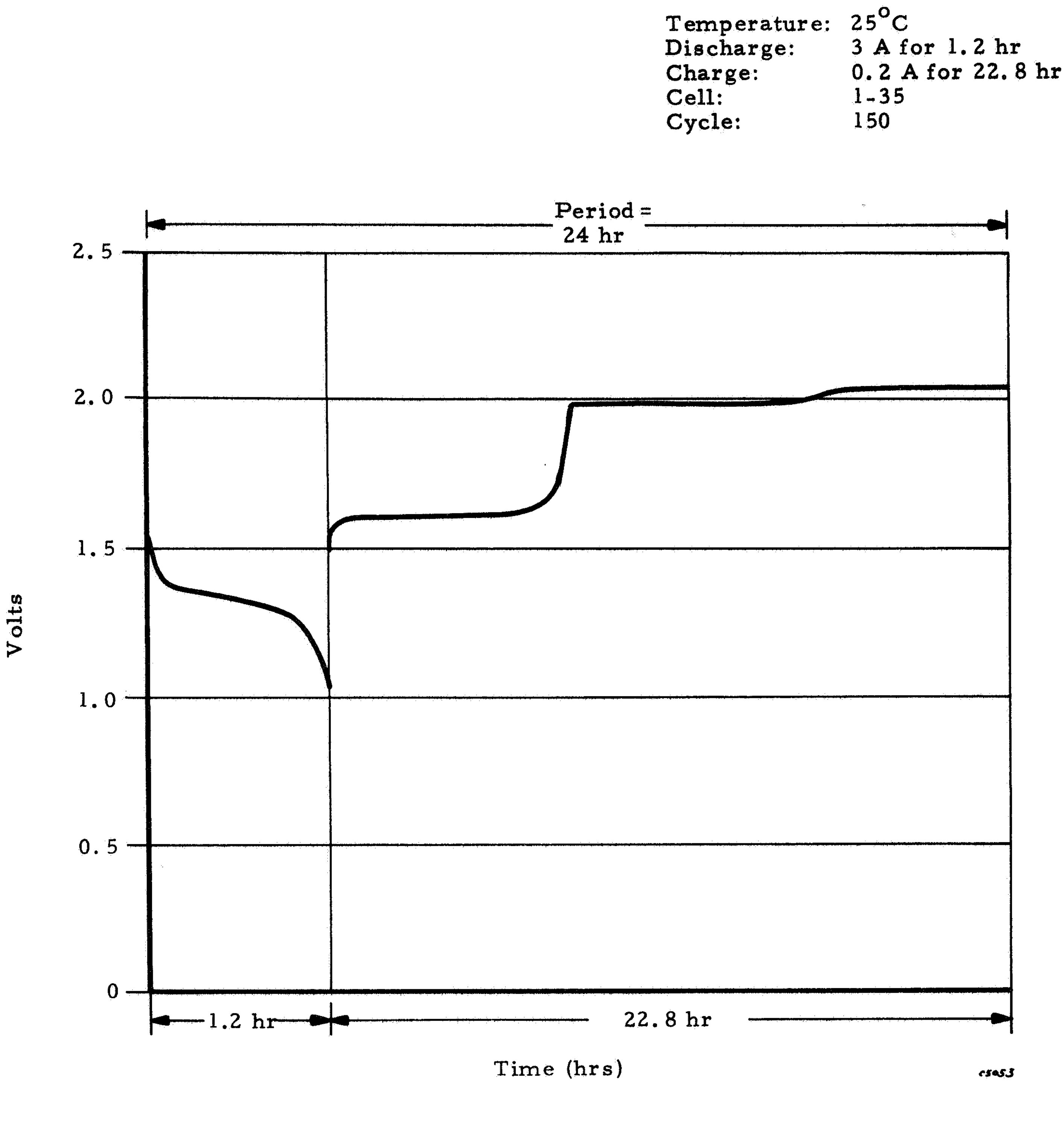


Figure 51. Cycling Curves for Lot 1 - Test C

Lot #2 — Test D	Tables LXIX-LXX	Figures 52-54
Test E	Tables LXXI-LXXV	Figures 55-56
Lot #3 — Test A	Tables LXXVI-LXXVII	Figures 57-62
Test B	Tables LXXVIII-LXXIX	Figures 63-64
Test C	Tables LXXX-LXXXI	
Lot #4 — Test D	Tables LXXXII-LXXXIII	Figure 65
Test E	Tables LXXXIV-LXXXV	Figure 66
Lot #5 — Test A	Tables LXXXVI-LXXXVII	Figures 67-69
Test B	Table LXXXVIII	Figure 70
Test C	Table LXXXIX	Figures 71-72
Lot #6 — Test D		Figures 73-77
Test E		Figures 78-79

Tables XC and XCI give the occurrence of the first three cycling failures of all cells for each test and each lot.

All cells showed the same type of failure: cracked separators caused by the swelling of the zinc electrode, more acute at the bottom. Some silver and zinc penetration was noticed in long cycle life cells. All other components appeared satisfactory: silver electrodes, separator edge seals, collars and leads.

Electrolyte addition was done at various times during the cycle life of the cells. For sake of convenience, the amounts added between arbitrarily fixed cycle numbers were lumped together and are reported in table form for each lot (Tables XCII to XCVII). The tables give also as a comparative figure the average amount used per 100 cycles over the total cycle life of the cells for each test condition. The average varies significantly with the test conditions: it is nil on the 24 hr-cycling period regime (Test C) where the cells are never overcharged; it is about 1 cc on a 1.5 hr-period regime (Test A) with low depth of discharge; it is about 1.5 to 2 cc on a 2 hr-period regime (Test D); it is in the range of 4 cc on a 2 hr-period regime with high rate (Test E), and it goes up to 9 cc on the 100^{o}C test regime (Test B).

The establishment of relationships between various magnitudes (depth of discharge, discharge current density, temperature and number of cycles) was

TABLE LXIX

UNIFORMITY STUDY, LOT #2, TEST D

Regime: Discharge: 3.0 A for 0.58 hr
Charge: 1.45 A for 1.42 hr
Voltage Limit: 2.08 V/cell
Temperature: 25°C

Cycle 1-10

Cell Number		26	27	28	29		Avg.
Charge	m%	43	43	43	43		43
(OC = 4%)	V_f	2.08	2.03	2.03	2.05		2.05
Discharge	p%	12	12	12	12		12
	V_p	1.29	1.33	1.32	1.32		1.32
	V_e	1.26	1.31	1.30	1.30		1.29
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

Cycle 300

Charge	m%	25	25	25	25		25
(OC = 5%)	V_f	2.0	2.14	2.02	2.15		2.08
Discharge	p%	25	25	25	25		25
	V_p	1.35	1.34	1.33	1.34		1.34
	V_e	1.30	1.28	1.28	1.29		1.28
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

TABLE LXX

UNIFORMITY STUDY, LOT #2, TEST D

Regime: Discharge: 3.0 A for 0.58 hr
Charge: 1.45 A for 1.42 hr
Voltage Limit: 2.08 V/cell
Temperature: 25 °C

Cycle 600*

Cell Number		26*	27*	28	29		Avg.
Charge	m%			25	27		26
(OC = 10 %)	V_f			2.10	2.06		2.08
Discharge	p%			32	37		35
	V_p			1.32	1.32		1.32
	V_e			1.24	1.24		1.24
Electrolyte Addition	Cum. Amt (cc)	0		3	3		3

*Cell #26 failed at 493 cycles.
Cell #27 failed at 453 cycles.

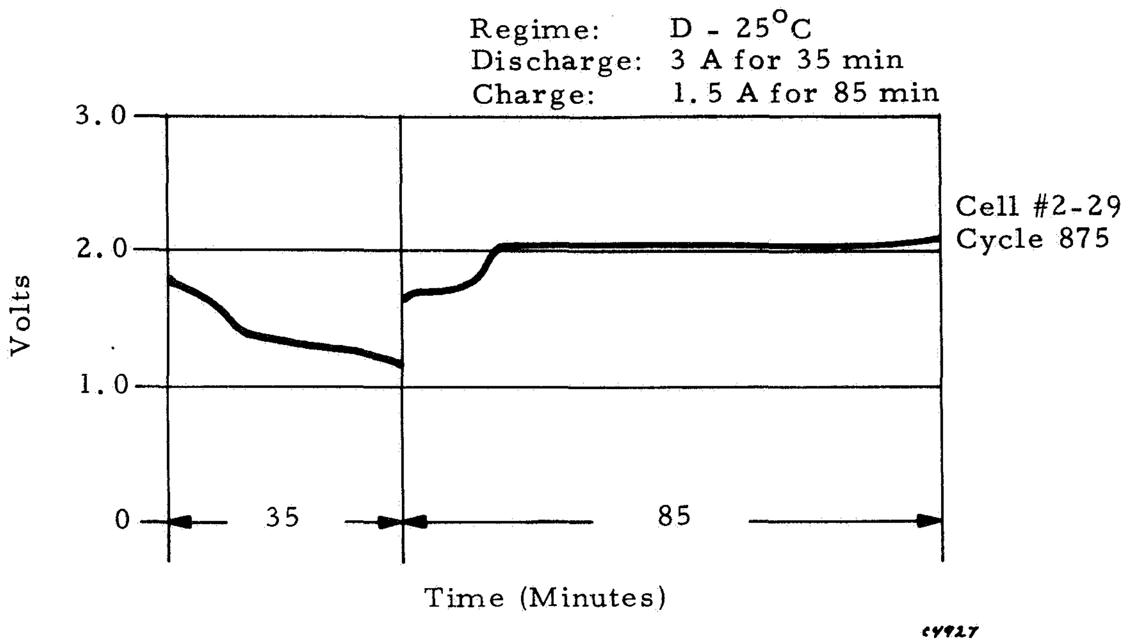


Figure 52. Cycling Curves, Lot #2 - Test D

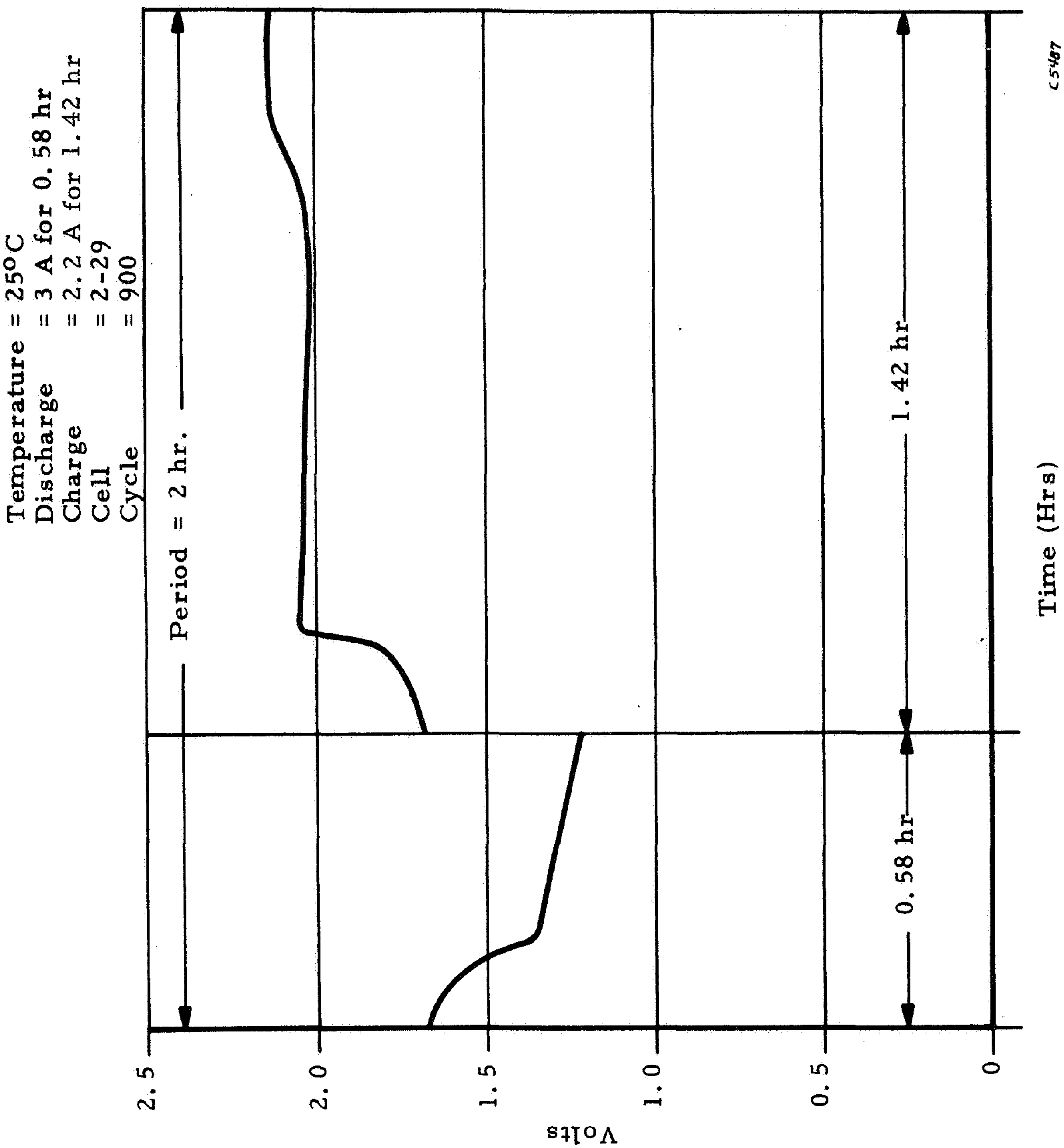


Figure 53. Cycling Curves, Lot #2 - Test D

Temperature: $25^{\circ}C$
Discharge: 3 A for 0.58 hr
Charge: 2.2 A for 1.42 hr
Cell: 2-28
Cycle: 1200

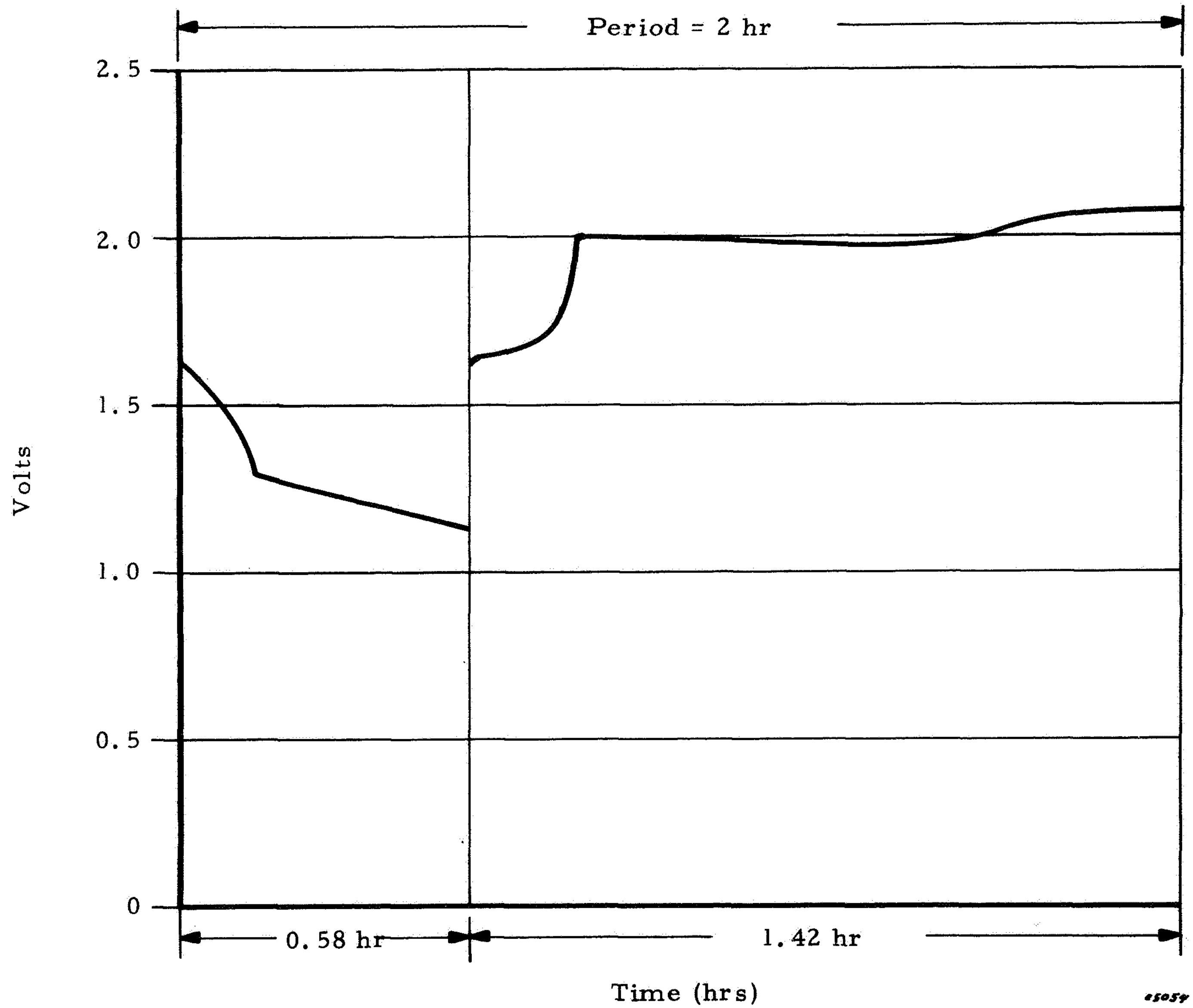


Figure 54. Cycling Curves For Lot 2 Test D

TABLE LXXI

UNIFORMITY STUDY, LOT #2, TEST E GROUP I (PRESSURIZED, 40% KOH)

Regime: Discharge: 5.4 A for 0.58 hr
Charge: 2.5 A for 1.42 hr
Voltage Limit: 2.10 V/cell
Temperature: 25°C

Cycle 1-10

Cell Number		30	31	32	33		Avg.
Charge	m%	45	45	45	45		45
(OC = -4%)	V_f	2.10	2.10	2.09	2.11		2.10
Discharge	p%	13	13	13	13		13
	V_p	1.26	1.25	1.25	1.26		1.26
	V_e	1.24	1.23	1.23	1.24		1.24
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

Cycle 36-40*

Charge	m%	46	46	46	46		46
(OC = -2%)	V_f	2.28	2.22	2.24	2.28		2.25
Discharge	p%	8	8	8	8		8
	V_p	1.30	1.30	1.22	1.28		1.28
	V_e	1.14	1.18	1.02	1.04		1.10
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

*Voltage limit was increased to 2.25 V/cell in order to overcome extreme undercharge condition.

TABLE LXXII

UNIFORMITY STUDY, LOT #2, TEST E
GROUP II (VENTED, 30% KOH)

Regime: Discharge: 5.4 A for 0.58 hr
Charge: 2.5 A for 1.42 hr
Voltage Limit: 2.10 V/cell
Temperature: 25°C

Cycle 1-10

Cell Number		21	22	23	24		Avg.
Charge	m%	29	29	29	29		29
(OC = 1.2%)	V_f	2.09	2.12	2.06	2.11		2.10
Discharge	p%	20	20	20	20		20
	V_p	1.32	1.33	1.33	1.31		1.32
	V_e	1.31	1.33	1.33	1.30		1.32
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

Cycle 40

Charge	m%	40	40	40	40		40
(OC = 3.7%)	V_f	2.10	2.10	2.09	2.10		2.10
Discharge	p%	21	21	21	21		21
	V_p	1.32	1.32	1.32	1.32		1.32
	V_e	1.28	1.28	1.28	1.27		1.28
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

TABLE LXXIII

UNIFORMITY STUDY, LOT #2, TEST E
GROUP II (VENTED, 30% KOH)

Regime: Discharge: 5.4 A for 0.58 hr
Charge: 2.5 A for 1.42 hr
Voltage Limit: 2.10 V/cell
Temperature: 25°C

Cycle 100

Cell Number		21	22	23	24		Avg.
Charge	m%	43	43	43	43		43
(OC = 7.5%)	V_f	2.08	2.12	2.12	2.08		2.10
Discharge	p%	22	22	22	22		22
	V_p	1.32	1.32	1.33	1.31		1.32
	V_e	1.31	1.30	1.34	1.30		1.31
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

TABLE LXXIV

UNIFORMITY STUDY, LOT #2, TEST E
GROUP III (VENTED, 40% KOH)

Regime: Discharge: 5.4 A for 0.58 hr
Charge: 2.5 A for 1.42 hr
Voltage Limit: 2.10 V/cell
Temperature: 25°C

Cycle 1-10

Cell Number		36	37	38			Avg.
Charge	m%	41	41	41			41
(OC = 1.0%)	V_f	2.12	2.11	2.09			2.10
Discharge	p%	14	14	14			14
	V_p	1.28	1.30	1.29			1.29
	V_e	1.27	1.30	1.28			1.28
Electrolyte Addition	Cum. Amt (cc)	0	0	0			0

Cycle 40*

Charge	m%	41	41	41			41
(OC = 2.3%)	V_f	2.16	2.15	2.14			2.15
Discharge	p%	14	14	14			14
	V_p	1.28	1.30	1.28			1.28
	V_e	1.26	1.29	1.27			1.27
Electrolyte Addition	Cum. Amt (cc)	0	0	0			0

*Voltage limit was increased to 2.15 in order to eliminate an undercharge condition.

TABLE LXXV

UNIFORMITY STUDY, LOT #2, TEST E
GROUP III (VENTED, 40% KOH

Regime: Discharge: 5.4 A for 0.58
Charge: 2.5 A for 1.42
Voltage Limit: 2.15 V/cell
Temperature: 25°C

Cycle 100

Cell Number		36	37	37			Avg.
Charge	m%	42	42	42			42
(OC = 1.0%)	V_f	2.14	2.11	2.16			2.14
Discharge	p%	16	16	16			16
	V_p	1.27	1.29	1.30			1.29
	V_e	1.20	1.19	1.19			1.19
Electrolyte Addition	Cum. Amt (cc)	0	0	0			0

Cycle 200

Charge	m%	38	38	38			38
(OC = 3.5%)	V_f	2.15	2.08	2.17			2.13
Discharge	p%	19	19	19			19
	V_p	1.23	1.26	1.28			1.26
	V_e	1.08	1.16	1.20			1.15
Electrolyte Addition	Cum. Amt (cc)	0	0	0			0

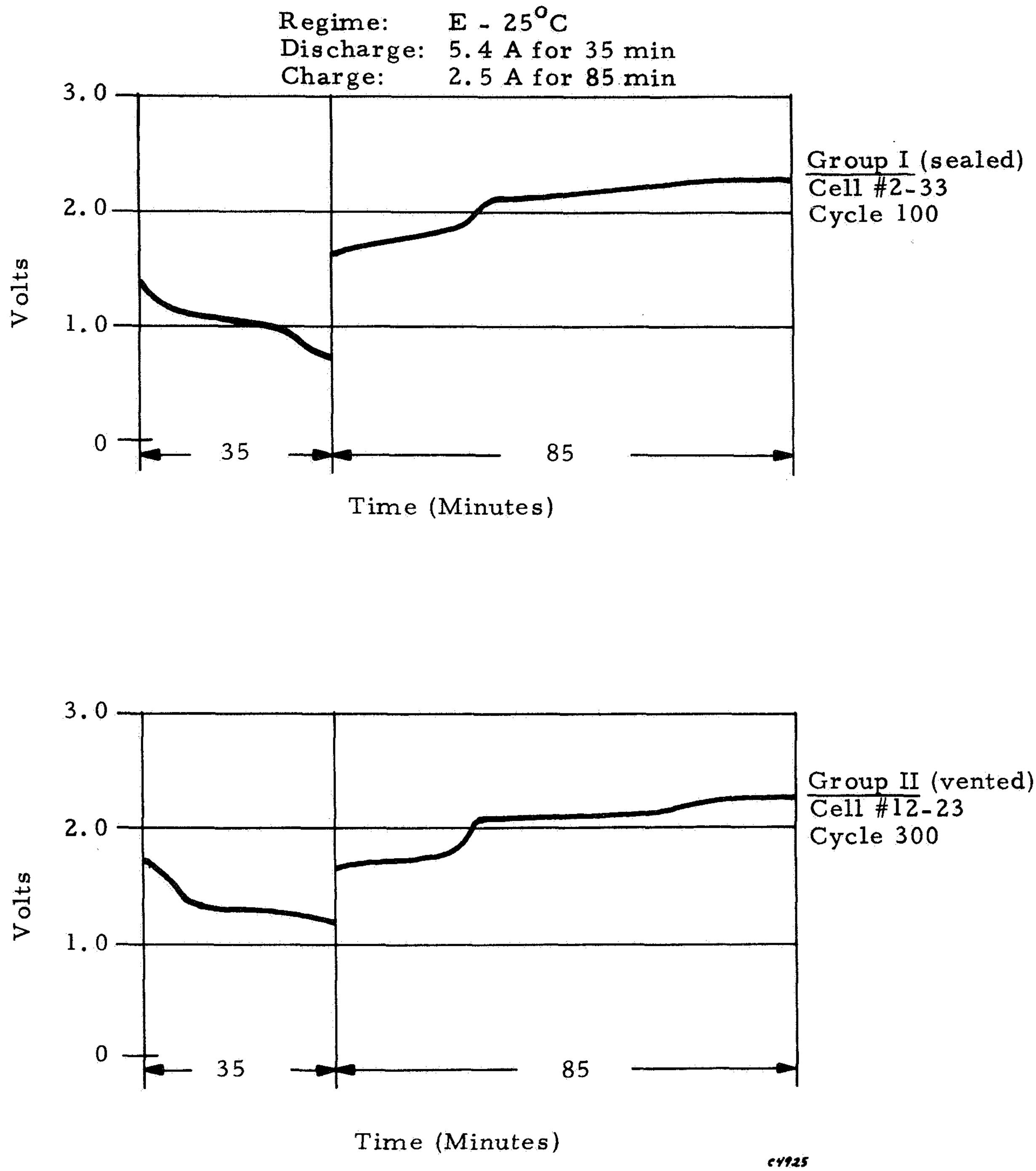


Figure 55. Cycling Curves, Lot #2 - Test E

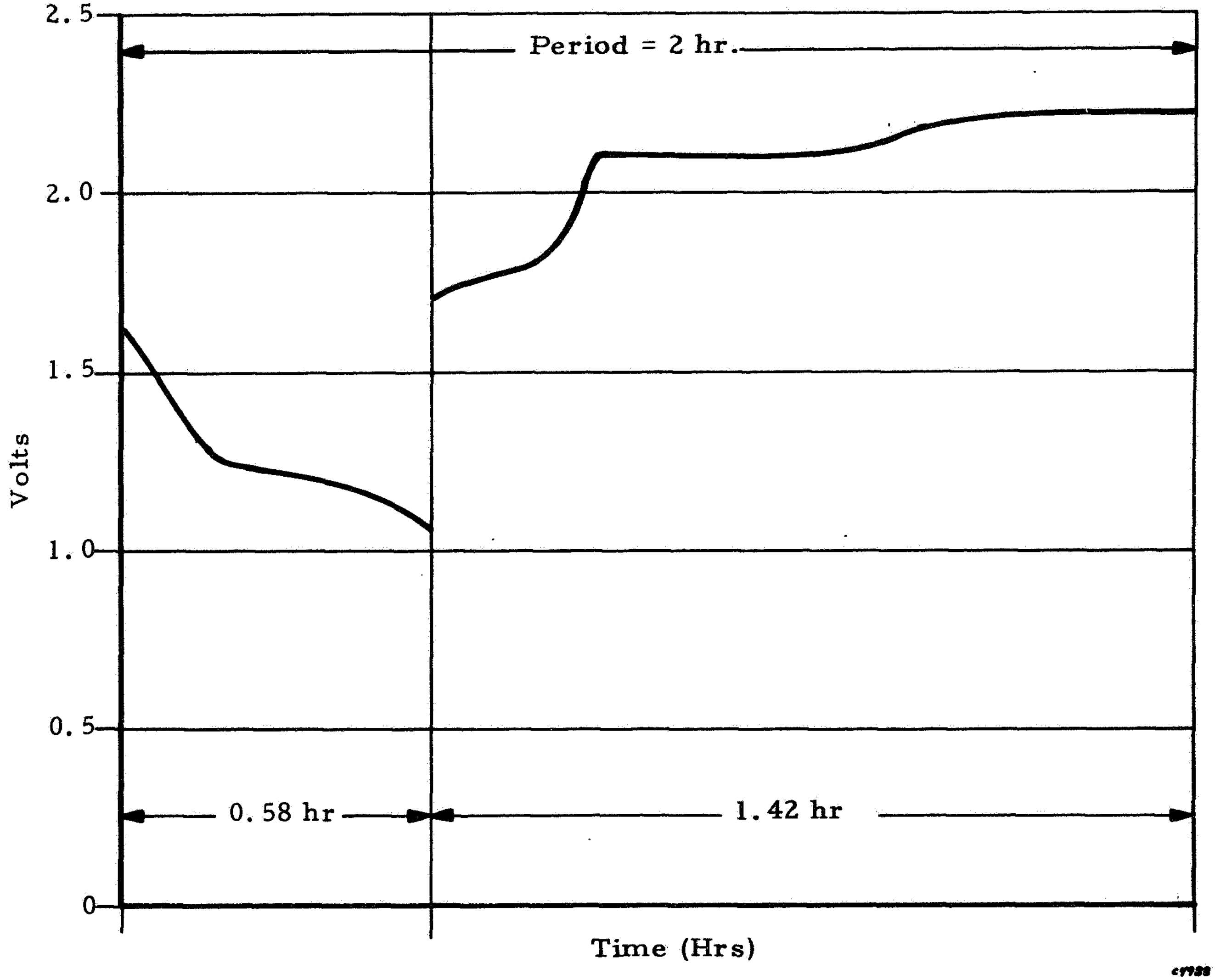


Figure 56. Cycling Curves, Lot #2 - Test E

TABLE LXXVI

UNIFORMITY STUDY, LOT #3, TEST A

Regime: Discharge: 2.0 A for 0.5 hr
Charge: 1.70 A for 1.0 hr
Voltage Limit: 2.10 V/cell
Temperature: 25 °C

Cycle 1-10

Cell Number		26	27	28	29	36	Avg.
Charge	m%	26	29	29	26	33	29
(OC = 4.8 %)	V_f	2.11	2.12	2.08	2.09	2.14	2.11
Discharge	p%	15	15	15	17	29	18
	V_p	1.36	1.34	1.34	1.36	1.33	1.34
	V_e	1.34	1.32	1.32	1.34	1.23	1.31
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0	0	0

Cycle 300

Charge	m%	40	33	33	*	33	35
(OC = 0 %)	V_f	2.14	2.04	2.11		2.20	2.12
Discharge	p%	24	24	24		26	25
	V_p	1.30	1.30	1.30		1.20	1.28
	V_e	1.21	1.25	1.22		1.12	1.20
Electrolyte Addition	Cum. Amt (cc)	0	0	0		0	0

*Cell #29 replaced by cell #36 when it failed at 243 cycles

TABLE LXXVII

UNIFORMITY STUDY,
LOT #3, TEST A

Regime: Discharge: 3.0 A for 0.5 hr
Charge: 1.70 A for 1.0 hr
Voltage Limit: 2.10 V/cell
Temperature: 25 °C

Cycle 600

Cell Number		26	27	28	36*		Avg.
Charge	m%	30	38	23			30
(OC = 17 %)	V_f	2.24	2.08	2.04			2.12
Discharge	p%	28	26	26			27
	V_p	1.30	1.24	1.30			1.28
	V_e	1.27	1.22	1.24			1.26
Electrolyte Addition	Cum. Amt (cc)	0	0	0			

*Cell #36 has not reached 600 cycles yet. It is 293 cycles behind the other 3 cells.

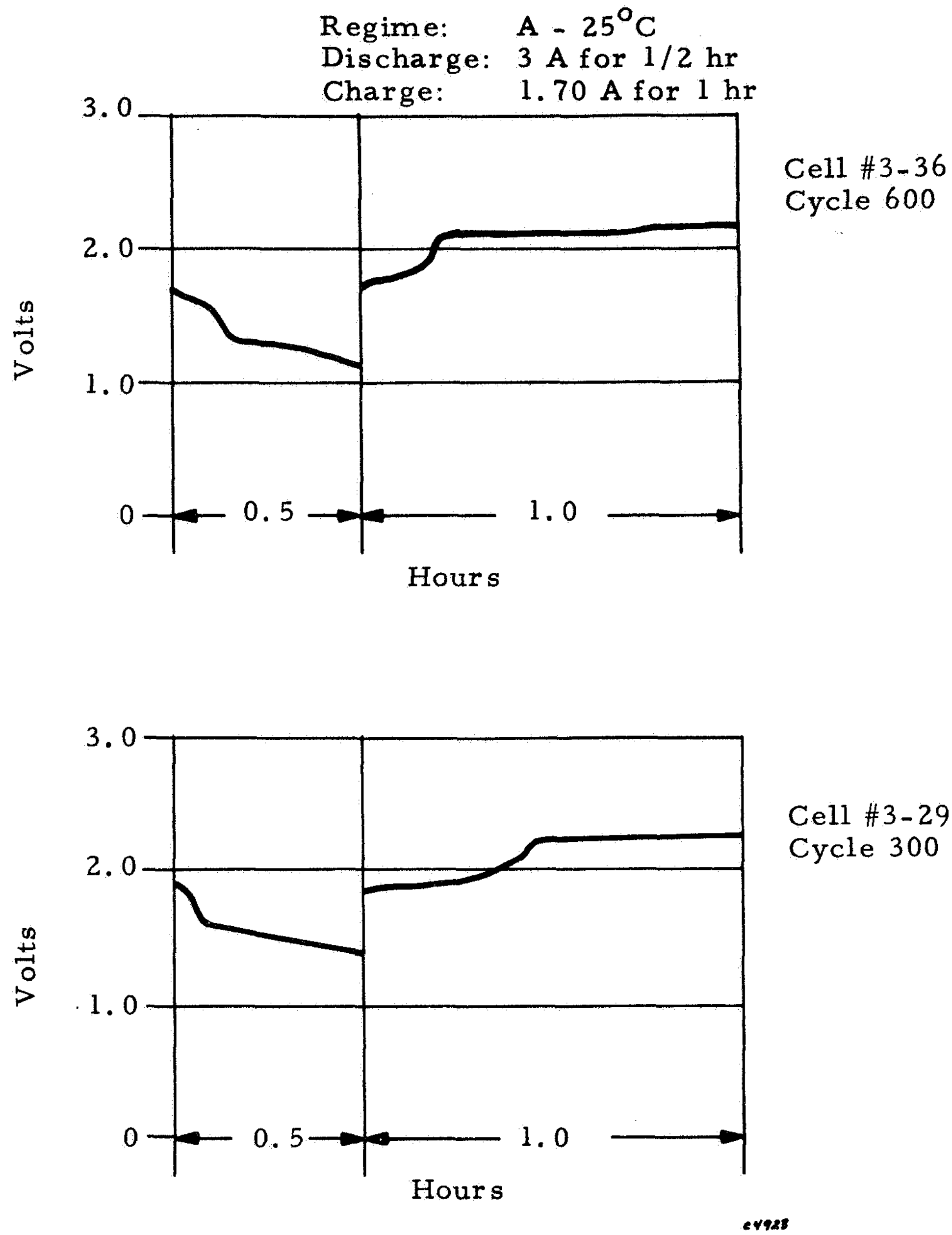


Figure 57. Cycling Curves, Lot #3 - Test A

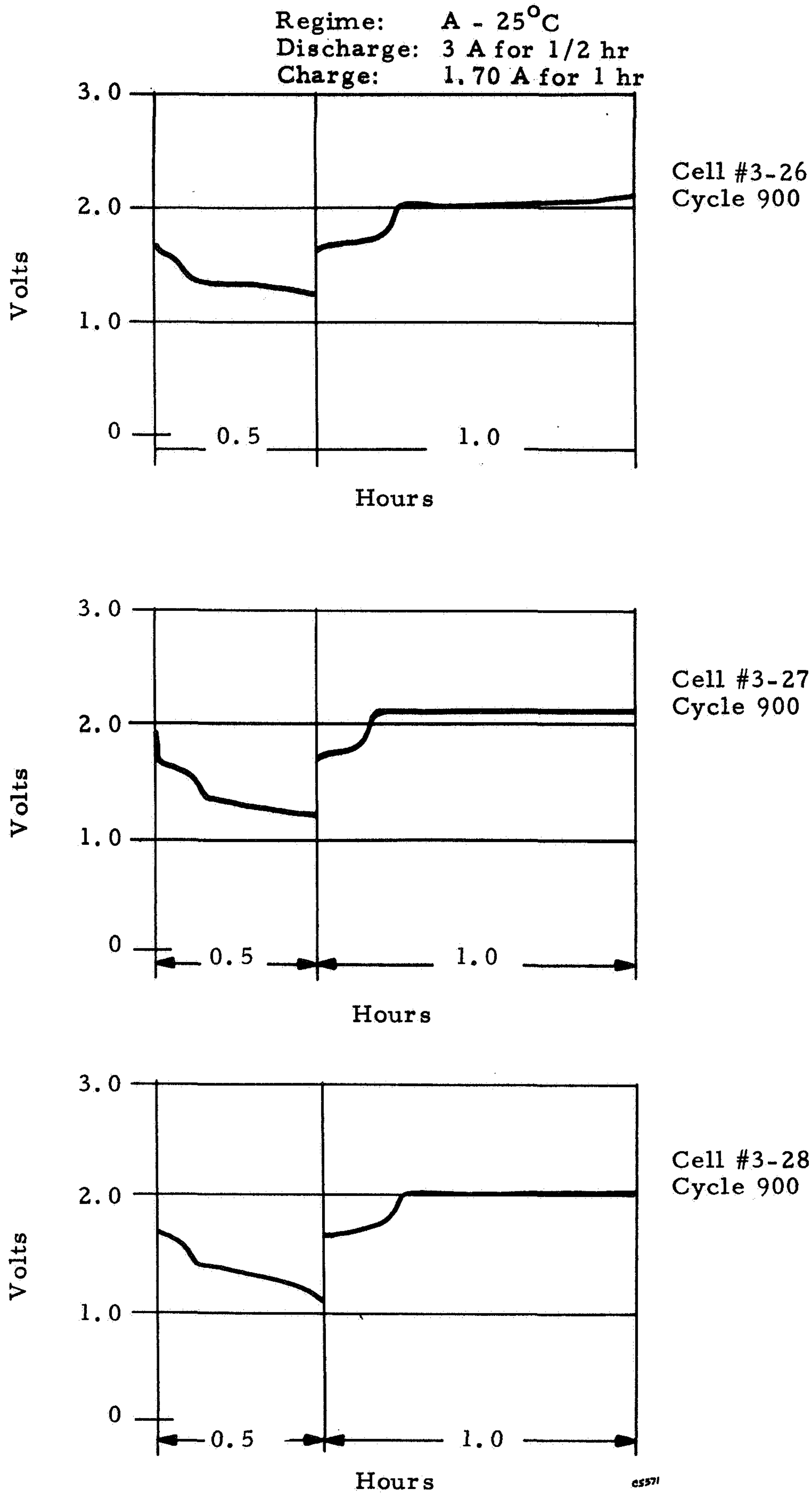


Figure 58. Cycling Curves, Lot #3 - Test A

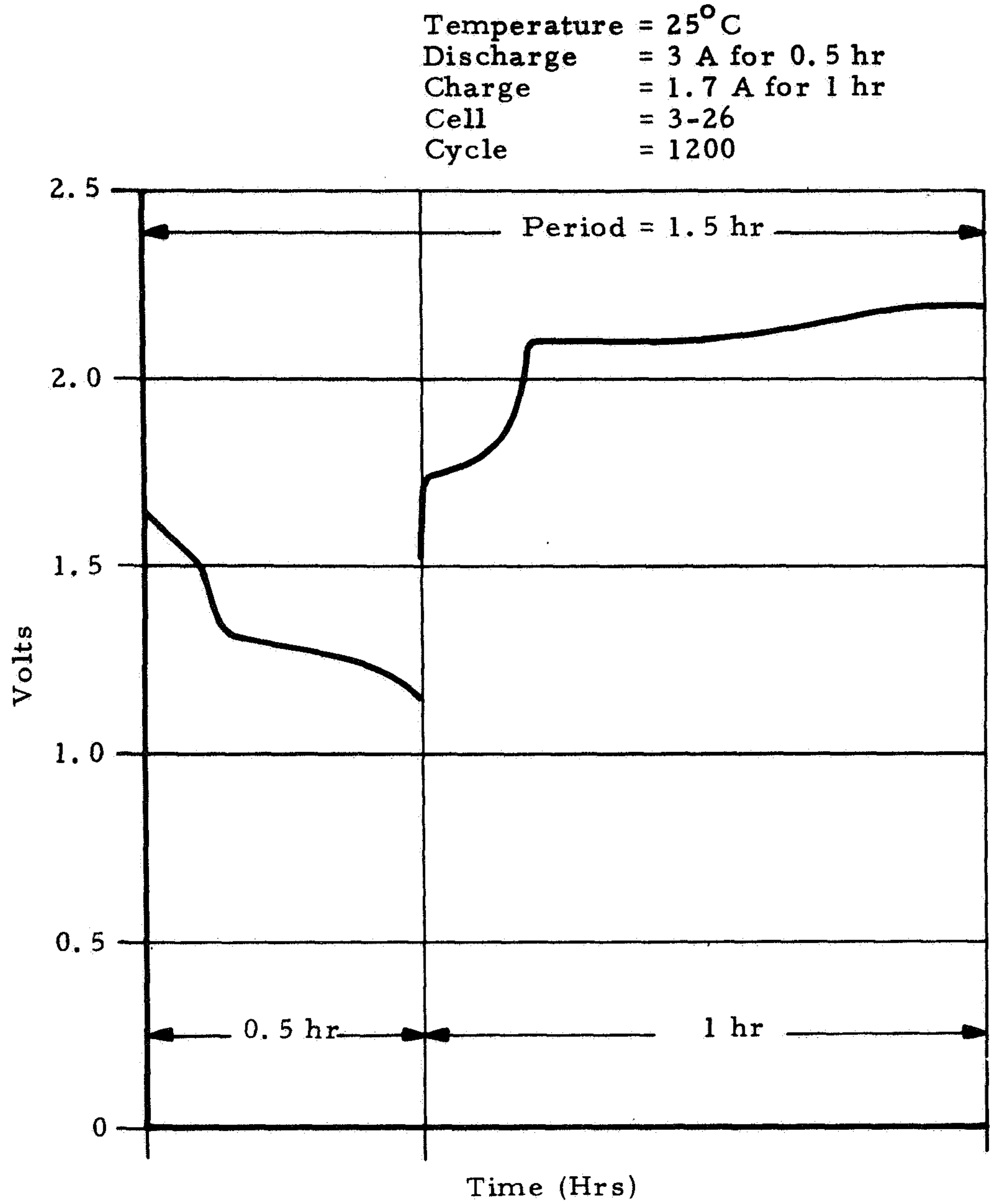


Figure 59. Cycling Curves, Lot #3 - Test A

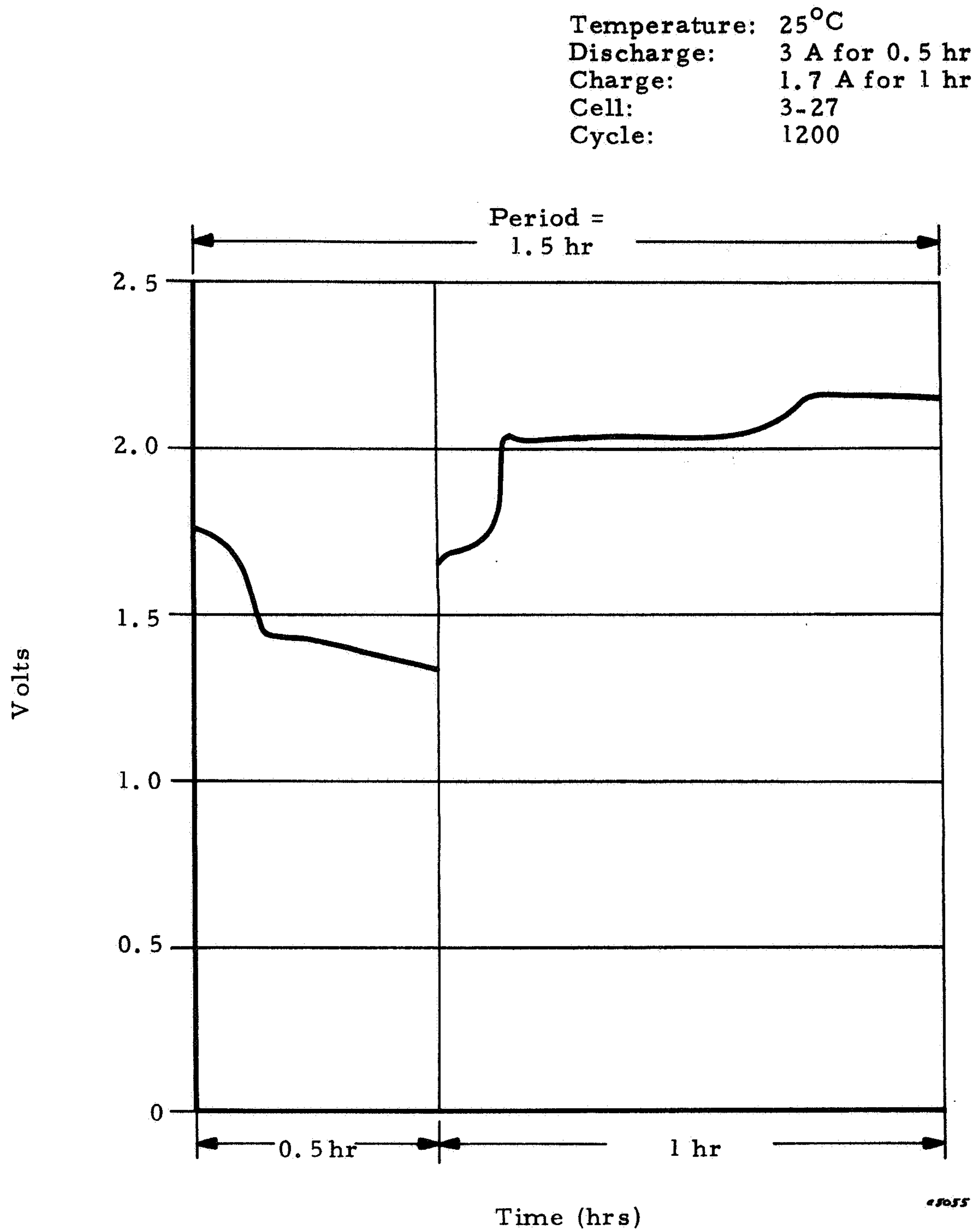


Figure 60. Cycling Curves for Lot 3 - Test A

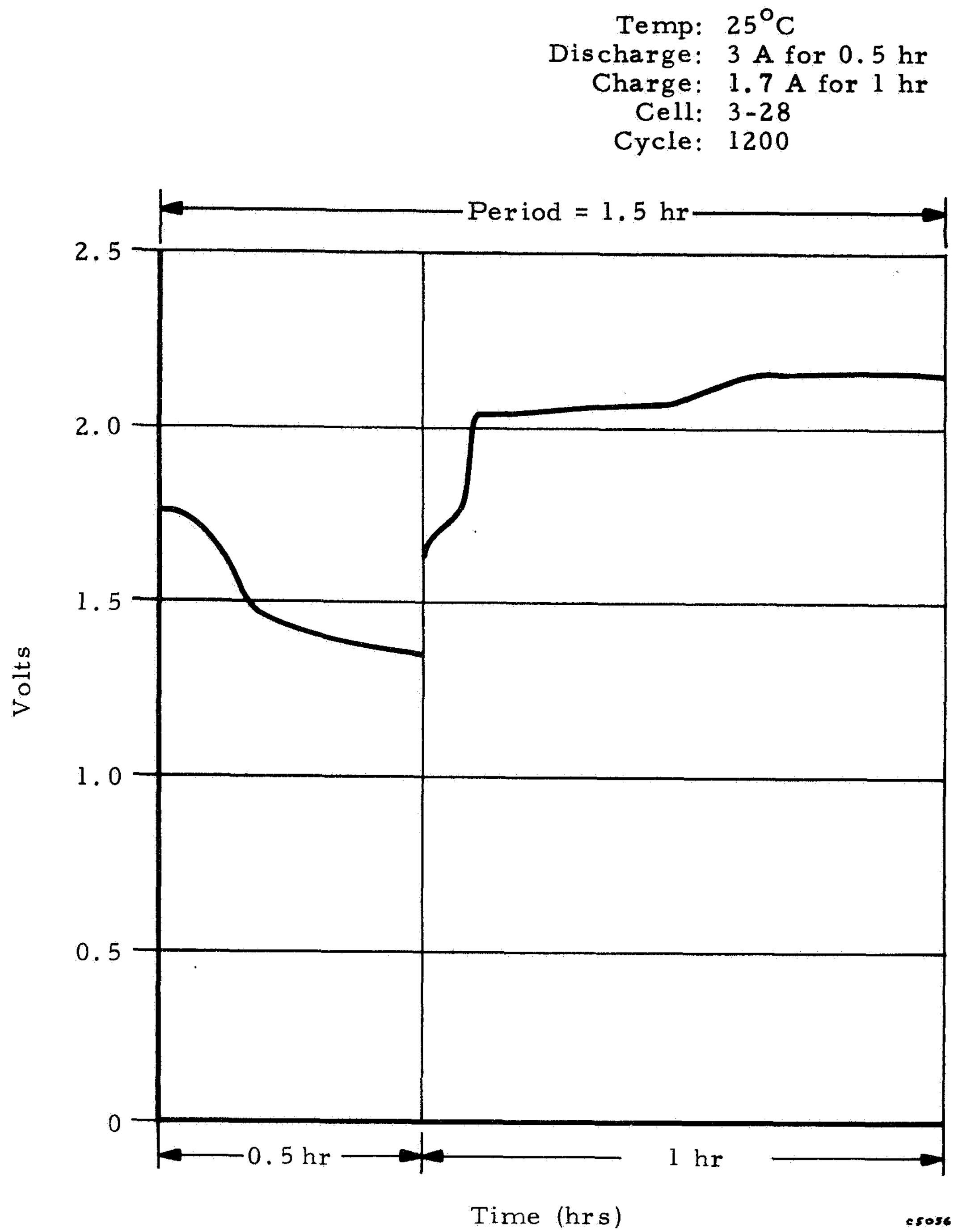


Figure 61. Cycling Curves for Lot 3 - Test A

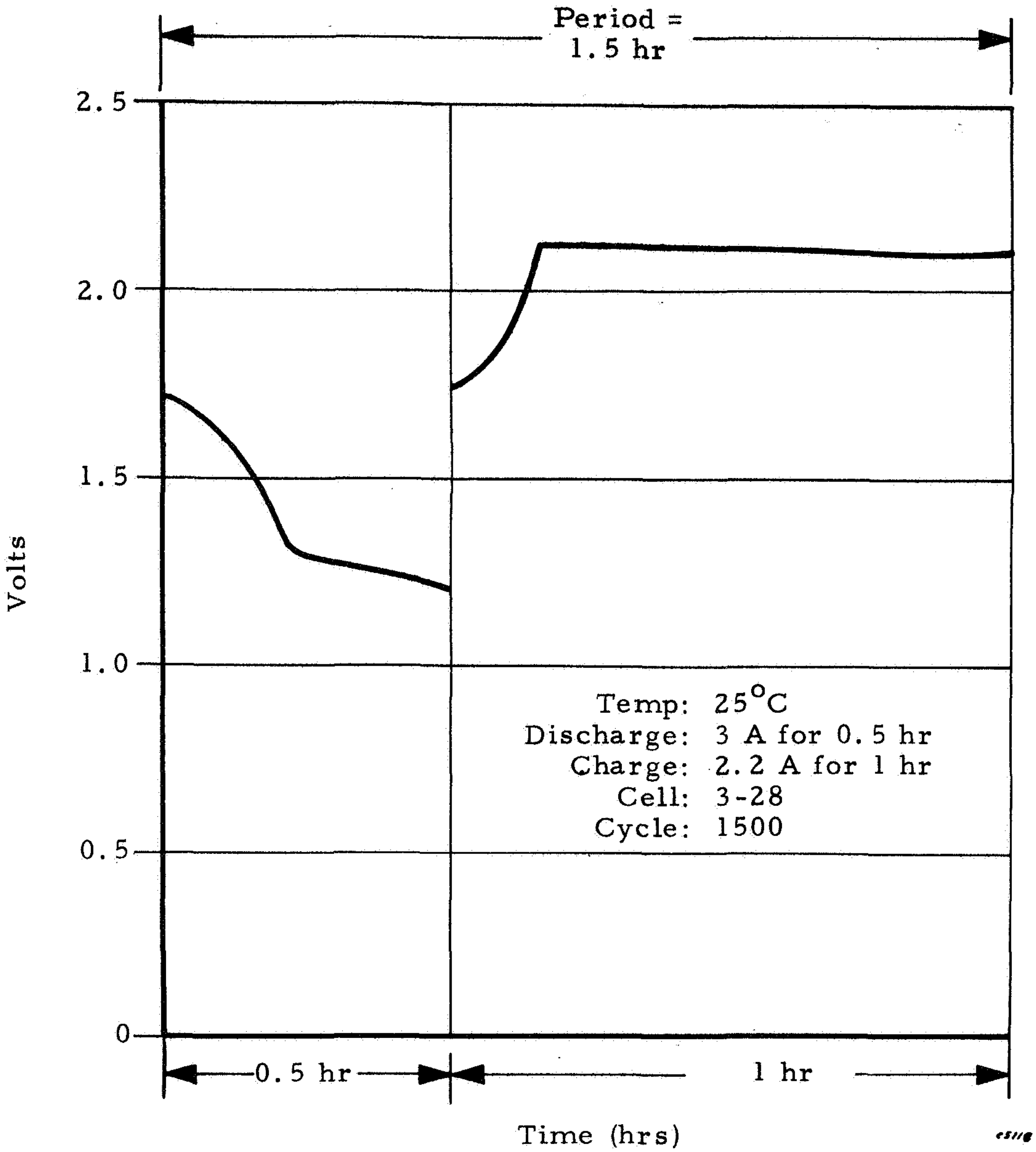


Figure 62. Cycling Curves for Lot 3 - Test A

TABLE LXXVIII

UNIFORMITY STUDY, LOT #3, TEST B

Regime: Discharge: 3.0 A for 0.5 hr
Charge: 1.60 A for 1 hr
Voltage Limit: 2.02 V/cell
Temperature: 100 °C

Cycle 1-10

Cell Number		30	31	32	33		Avg.
Charge	m%	37	32	37	32		35
(OC = 8.1 %)	V_f	2.03	2.00	2.02	2.02		2.02
Discharge	p%	14	14	14	14		14
	V_p	1.40	1.41	1.41	1.40		1.41
	V_e	1.40	1.41	1.41	1.40		1.41
Electrolyte Addition	Cum. Amt (cc)	0	0	1	1		1

Cycle 100

Charge	m%	33	33	33	83		45
(OC = 9.5 %)	V_f	2.06	2.03	2.01	1.96		2.02
Discharge	p%	17	17	17	2		13
	V_p	1.43	1.43	1.43	1.43		1.43
	V_e	1.41	1.41	1.41	1.33		1.39
Electrolyte Addition	Cum. Amt (cc)	14	11	13	13		13

TABLE LXXIX

UNIFORMITY STUDY,
LOT #3, TEST B

Regime: Discharge: 3.0 A for 0.5 hr
Charge: 1.60 A for 1 hr
Voltage Limit: 2.02 V/cell
Temperature: 100 °C

Cycle 200

Cell Number		30	31*	32	33*		Avg.
Charge	m%	37	60	37			45
(OC = 6 %)	V_f	2.03	2.00	2.02			2.02
Discharge	p%	21	12	21			18
	V_p	1.41	1.41	1.41			1.41
	V_e	1.39	1.31	1.39			1.36
Electrolyte Addition	Cum. Amt (cc)	31	27	30			29

Cycle 300

Charge	m%	40	*	42			41
(OC = 8 %)	V_f	2.00		2.08			2.04
Discharge	p%	22		24			23
	V_p	1.38		1.36			1.37
	V_e	1.20		1.24			1.22
Electrolyte Addition	Cum. Amt (cc)	42		44			43

*Cell #33 failed at 112 cycles and cell #31 at 225 cycles

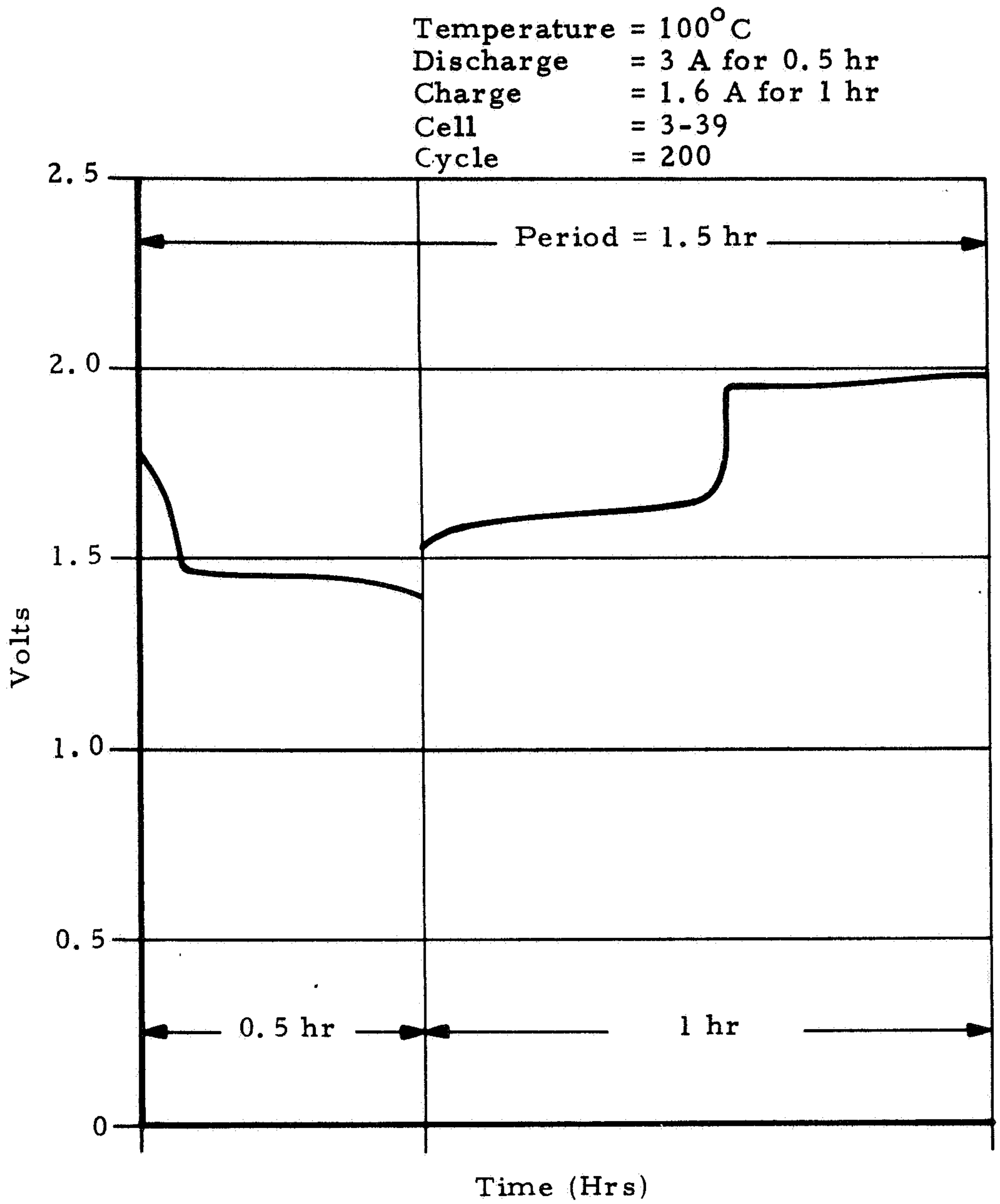


Figure 63. Cycling Curves, Lot #3 - Test B

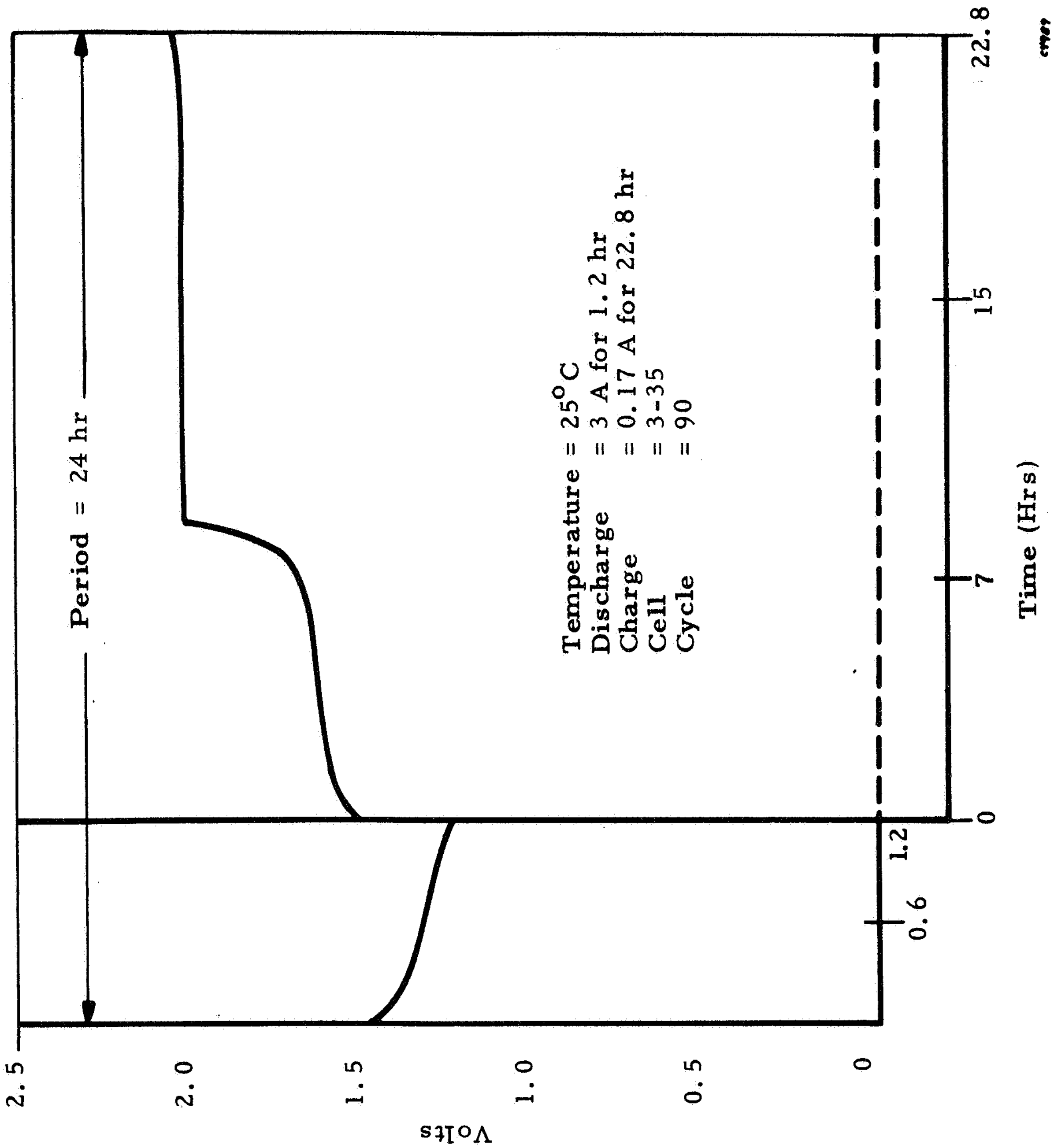


Figure 64. Cycling Curves, Lot #3 - Test C

TABLE LXXX

UNIFORMITY STUDY, LOT #3, TEST C

Regime: Discharge: 3.0 A for 1.2 hr
Charge: 0.170 A for 22.8 hr
Voltage Limit: 2.04 V/cell
Temperature: 25 °C

Cycle 1

Cell Number		34	35				Avg.
Charge	m%	30	30				30
(OC = 11.8 %)	V_f	1.99	1.99				1.99
Discharge	p%	5	5				5
	V_p	1.36	1.36				1.36
	V_e	1.34	1.35				1.35
Electrolyte Addition	Cum. Amt (cc)	0	0				0

Cycle 30

Charge	m%	39	39				39
(OC = 10 %)	V_f	2.04	2.05				2.05
Discharge	p%	3	3				3
	V_p	1.30	1.30				1.30
	V_e	1.28	1.27				1.28
Electrolyte Addition	Cum. Amt (cc)	0	0				0

TABLE LXXXI

UNIFORMITY STUDY, LOT #3, TEST C

Regime: Discharge: 3.0 A for 1.2 hr

Charge: 0.170 A for 22.8 hr

Voltage Limit: 2.04 V/cell

Temperature: 25 °C

Cycle 60

Cell Number		34	35				Avg.
Charge	m%	37	14				26
(OC = 5.4 %)	V_f	1.96*	2.08*				2.04
Discharge	p%	5	10				8
	V_p	1.30	1.42				1.36
	V_e	1.20	1.40				1.30
Electrolyte Addition	Cum. Amt (cc)	0	0				0

*Note the non-uniformity.

TABLE LXXXII

UNIFORMITY STUDY, LOT #4, TEST D

Regime: Discharge: 3.0 A for .58 hr
Charge: 1.45 A for 1.42 hr
Voltage Limit: 2.04 V/cell
Temperature: 25 °C

Cycle 1-10

Cell Number		26	27	28	29		Avg.
Charge	m%	38	39	38	38		38
(OC = -.7 %)	V_f	2.08	2.08	2.08	2.08		2.08
Discharge	p%	19	19	19	19		19
	V_p	1.33	1.33	1.32	1.32		1.33
	V_e	1.28	1.28	1.28	1.28		1.28
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		

Cycle 300

Charge	m%	16	100	16	18		38
(OC = 30 %)	V_f	2.13	1.70*	2.15	2.15		2.03
Discharge	p%	26	0	26	26		20
	V_p	1.37	1.30	1.33	1.33		1.33
	V_e	1.29	1.11	1.26	1.25		1.23
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		

Cell #27 removed for a maintenance cycle.

TABLE LXXXIII

UNIFORMITY STUDY, LOT #4, TEST D

Regime: Discharge: 3.0 A for 0.58 hr
Charge: 1.80 A for 1.42 hr
Voltage Limit: 1.58 V/cell
Temperature: 25 °C

Cycle 600

Cell Number		26	27	28	29		Avg.
Charge	m%	13	24	12	14		16
(OC = 29 %)	V_f	2.12	1.95	2.09	2.11		2.07
Discharge	p%	25	12	29	25		23
	V_p	1.29	1.30	1.29	1.29		1.29
	V_e	1.21	1.20	1.21	1.20		1.21
Electrolyte Addition	Cum. Amt (cc)	3	2	4	0		2.25

*Voltage and current raised to get proper overcharge for continuous cycling.

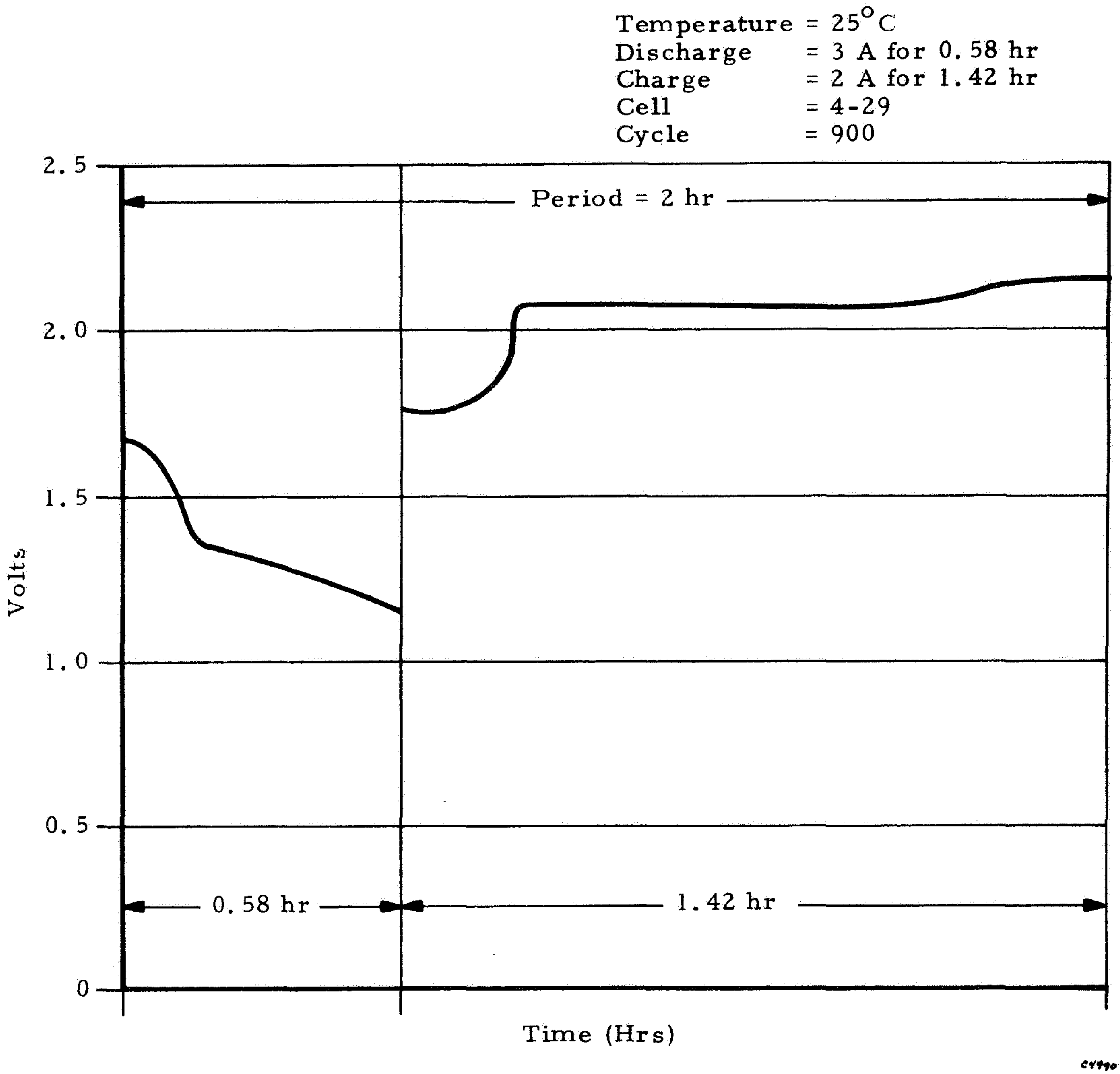


Figure 65. Cycling Curves, Lot #4 - Test D

TABLE LXXXIV

UNIFORMITY STUDY, LOT #4, TEST E

Regime: Discharge: 5.4 A for .58 hr
Charge: 2.5 A for 1.42 hr
Voltage Limit: 2.10 V/cell
Temperature: 25 °C

Cycle 1-10

Cell Number		30	31	32	33		Avg.
Charge	m%	44	44	45	45		45
(OC = 3.5 %)	V_f	2.09	2.09	2.10	2.11		2.10
Discharge	p%	18	18	18	18		18
	V_p	1.27	1.28	1.28	1.27		1.28
	V_e	1.20	1.22	1.23	1.21		1.21
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

Cycle 50*

Charge	m%	40	40	43	43		42
(OC = -1.1 %)	V_f	2.17	2.14	2.15	2.15		2.15
Discharge	p%	20	20	17	17		19
	V_p	1.25	1.28	1.23	1.22		1.25
	V_e	1.14	1.17	1.03	1.00		1.09
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

*V_f raised to 2.15 v/cell to try to get greater overcharge

TABLE LXXXV

UNIFORMITY STUDY, LOT #4, TEST E

Regime: Discharge: 5.4 A for 0.58 hr
Charge: 2.5 A for 1.42 hr
Voltage Limit: 2.15 V/cell
Temperature: 25 °C

Cycle 100

Cell Number		1	2	3	4	5	Avg.
Charge	m%	37	37	37	37		37
(OC = 7.6 %)	V_f	2.16	2.14	2.09	2.18		2.14
Discharge	p%	14	14	14	14		14
	V_p	1.23	1.25	1.24	1.22		1.23
	V_e	1.15	1.15	1.02	1.01		1.08
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		

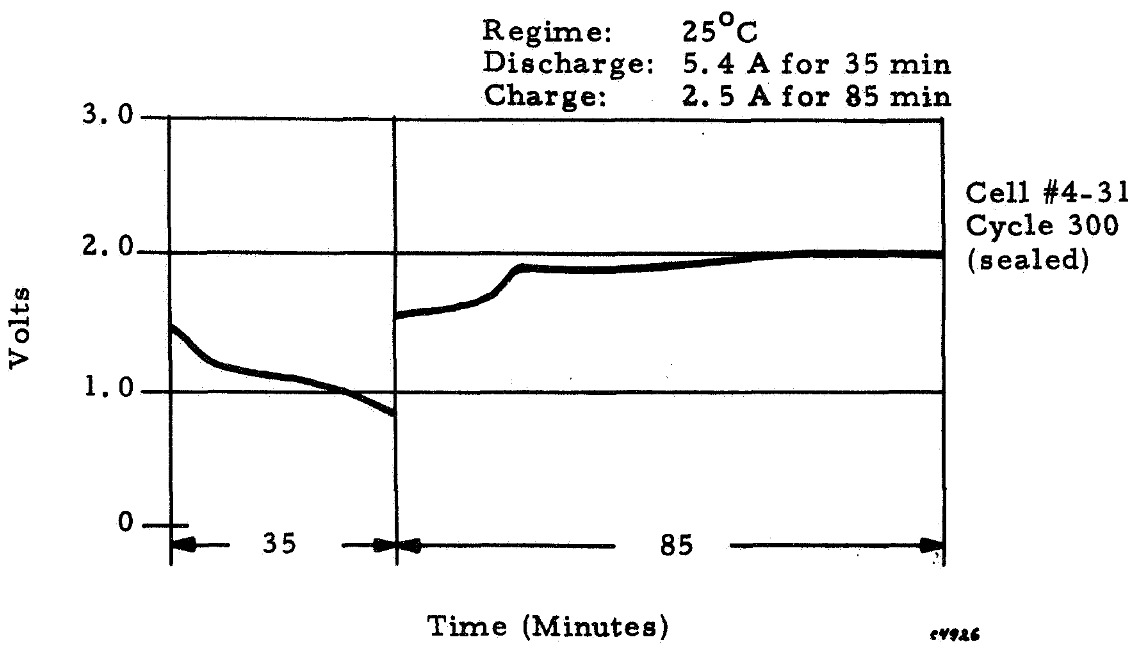


Figure 66. Cycling Curves, Lot #4 - Test E

TABLE LXXXVI

UNIFORMITY STUDY, LOT #5, TEST A

Regime: Discharge: 3.0 A for 0.5 hr
Charge: 1.60 A for 1.0 hr
Voltage Limit: 2.05 V/cell
Temperature: 25 °C

Cycle 1-10

Cell Number		26	27	28	29		Avg.
Charge	m%	40	43	40	43		41.5
(OC = -17 %)	V_f	2.05	2.05	2.05	2.05		2.05
Discharge	p%	18	18	18	18		18
	V_p	1.32	1.34	1.34	1.34		1.34
	V_e	1.30	1.32	1.31	1.32		1.31
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

Cycle 300

Charge	m%	29	29	100	25		46
(OC = 6 %)	V_f	2.15	2.27	1.71+	2.17		2.08
Discharge	p%	25	25	0	25		19
	V_p	1.34	1.35	1.32	1.32		1.32
	V_e	1.28	1.20	1.02	1.21		1.18
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

* Voltage limit raised to 2.08 V/cell average.
\+ Note incipient failure; cell reconditioned and put back on cycling.

TABLE LXXXVII

UNIFORMITY STUDY, LOT #5, TEST A

Regime: Discharge: 3.0 A for 0.5 hr

Charge: 1.8 A for 1.0 hr

Voltage Limit: 2.08 V/cell

Temperature: 25 °C

Cycle 600

Cell Number		26	27	28	29		Avg.
Charge	m%	20	20	40	20		25
(OC = 1.6 %)	V_f	2.11	2.11	1.97	2.14		2.08
Discharge	p%	28	28	15	28		25
	V_p	1.29	1.29	1.32	1.33		1.30
	V_e	1.22	1.28	1.22	1.27		1.25
Electrolyte Addition	Cum. Amt (cc)	0	0	1	0		0.25

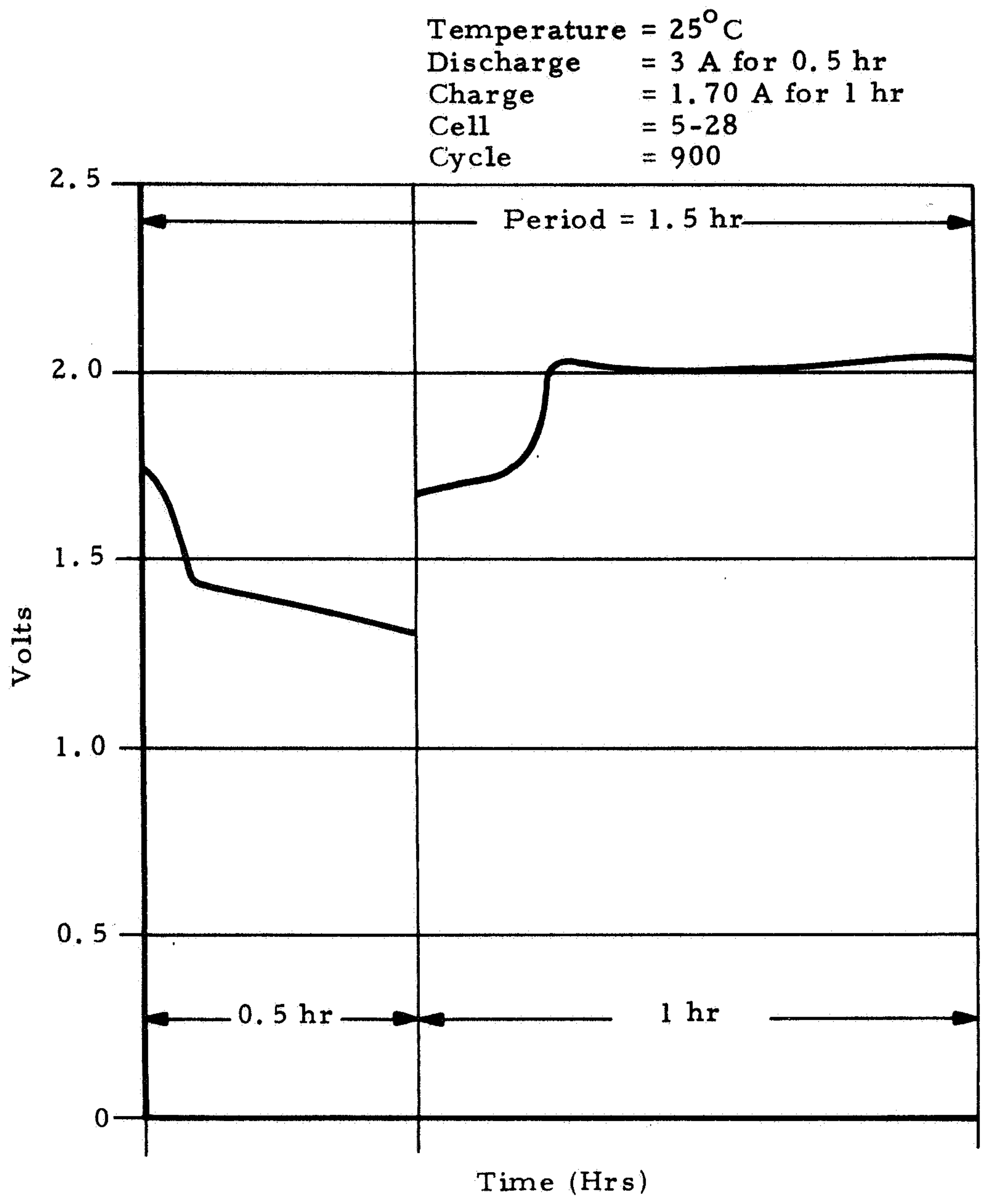


Figure 67. Cycling Curves, Lot #5 - Test A

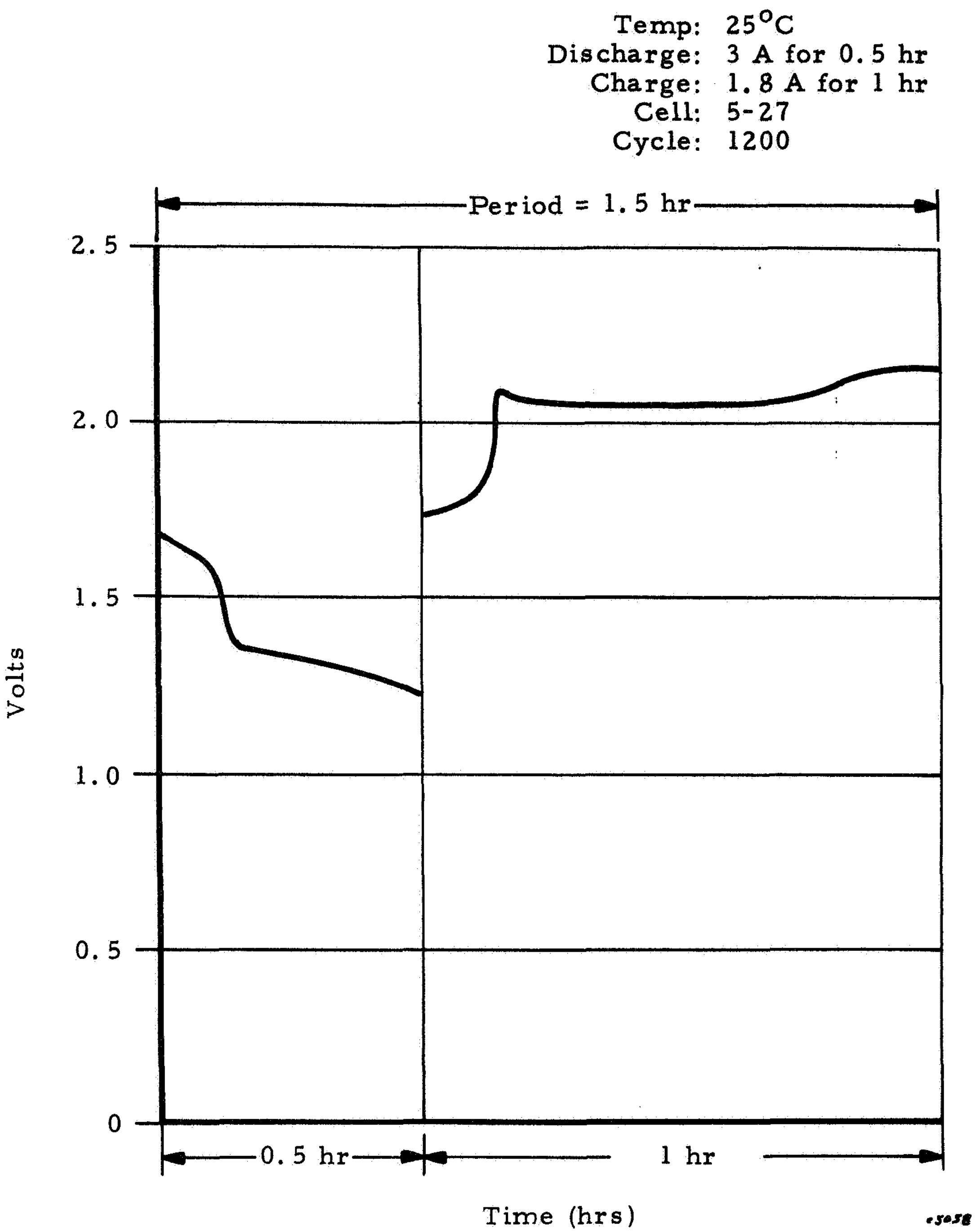


Figure 68. Cycling Curves for Lot #5 - Test A

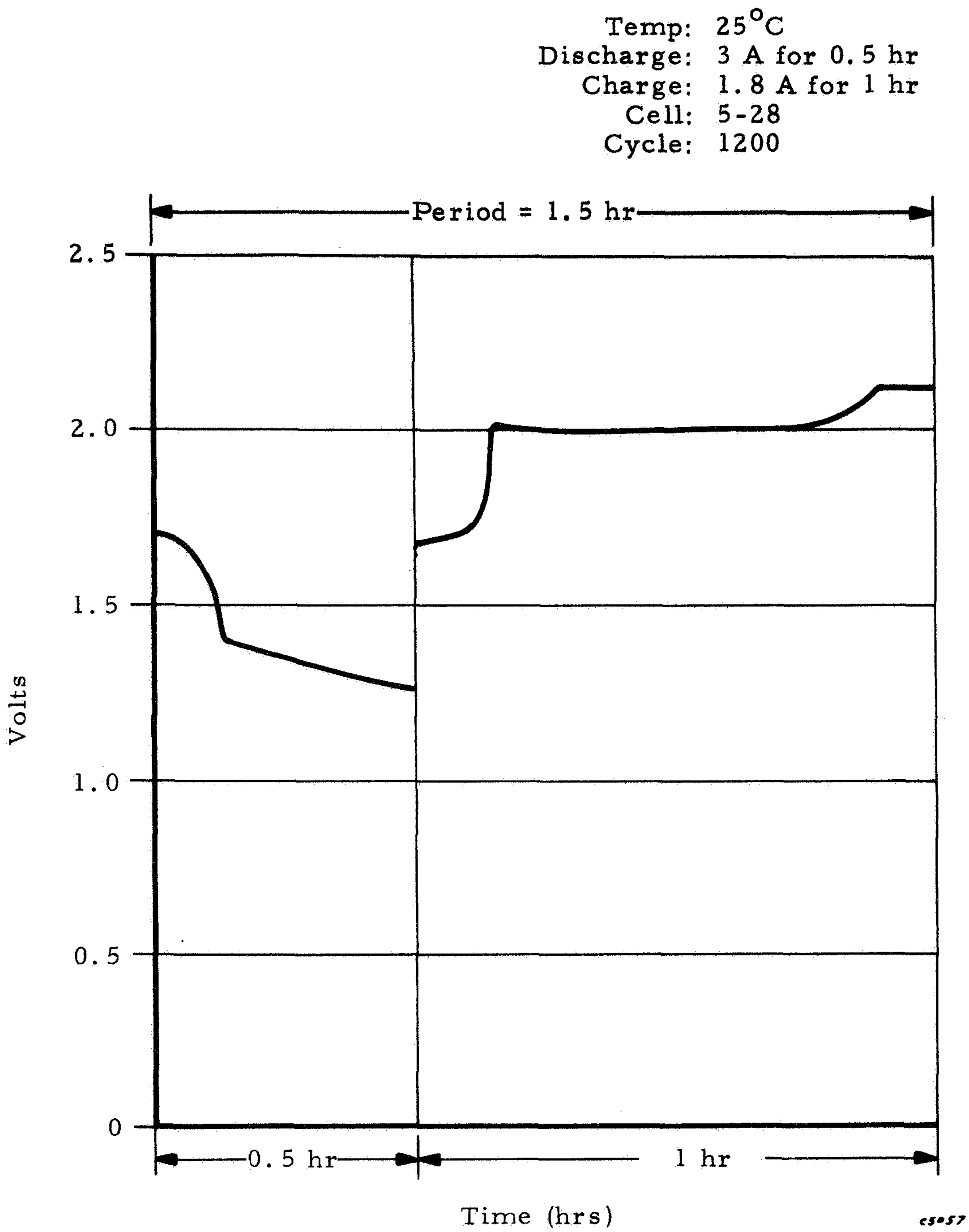


Figure 69. Cycling Curves for Lot #5 - Test A

TABLE LXXXVIII

UNIFORMITY STUDY, LOT #5, TEST B

Regime: Discharge: 3.0 A for 0.5 hr
Charge: 1.60 A for 1.0 hr
Voltage Limit: 2.03 V/cell
Temperature: 100 °C

Cycle 1-10

Cell Number		30	31	32	33		Avg.
Charge	m%	33	31	33	33		33
(OC = 2.9 %)	V_f	2.03	2.01	2.00	2.02		2.01
Discharge	p%	18	18	18	18		18
	V_p	1.47	1.46	1.47	1.47		1.47
	V_e	1.47	1.46	1.47	1.47		1.47
Electrolyte Addition	Cum. Amt (cc)	0	0	0	0		0

Cycle 200

Charge	m%	27	27	25	27		27
(OC = 4 %)	V_f	2.01	2.00	2.00	2.01		2.01
Discharge	p%	22	22	40	22		27
	V_p	1.46	1.44	1.46	1.44		1.45
	V_e	1.43	1.43	1.46	1.43		1.44
Electrolyte Addition	Cum. Amt (cc)	84	43	44	87		67

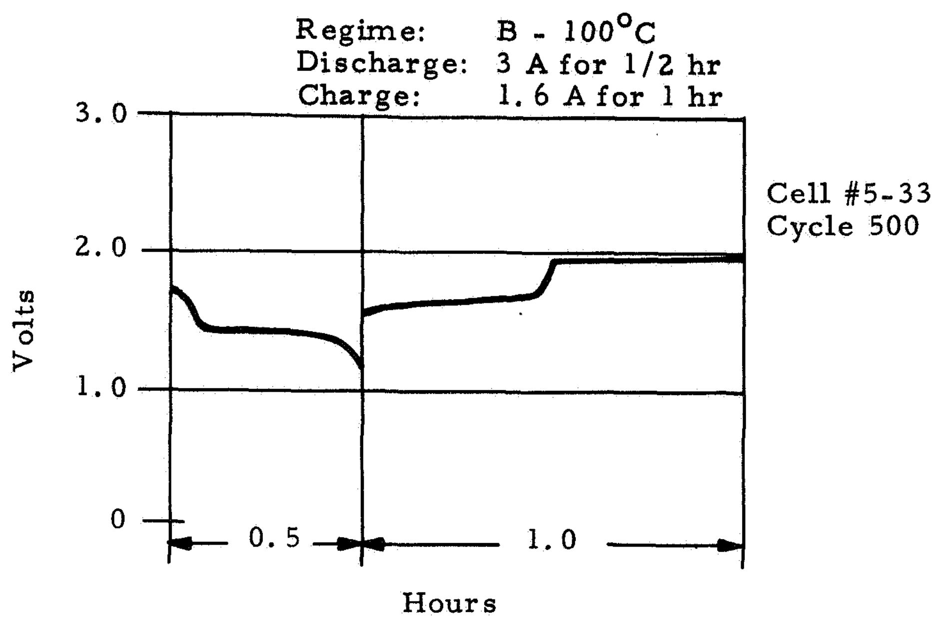


Figure 70. Cycling Curves, Lot #5 - Test B

TABLE LXXXIX

UNIFORMITY STUDY, LOT #5, TEST C

Regime: Discharge: 3.0 A for 1.2 hr
Charge: 0.170 A for 22.8 hr
Voltage Limit: 2.04 V/cell
Temperature: 25 °C

Cycle 1-10

Cell Number		34	35				Avg.
Charge	m%	1	1				1
(OC = 0.5 %)	V_f	2.04	2.02				2.03
Discharge	p%	4	4				4
	V_p	1.38	1.35				1.37
	V_e	1.38	1.35				1.37
Electrolyte Addition	Cum. Amt (cc)	0	0				0

Cycle 30

Charge	m%	25	25				25
(OC = 5 %)	V_f	2.02	2.04				2.03
Discharge	p%	2	4				3
	V_p	1.34	1.36				1.35
	V_e	1.28	1.30				1.29
Electrolyte Addition	Cum. Amt (cc)	0	0				0

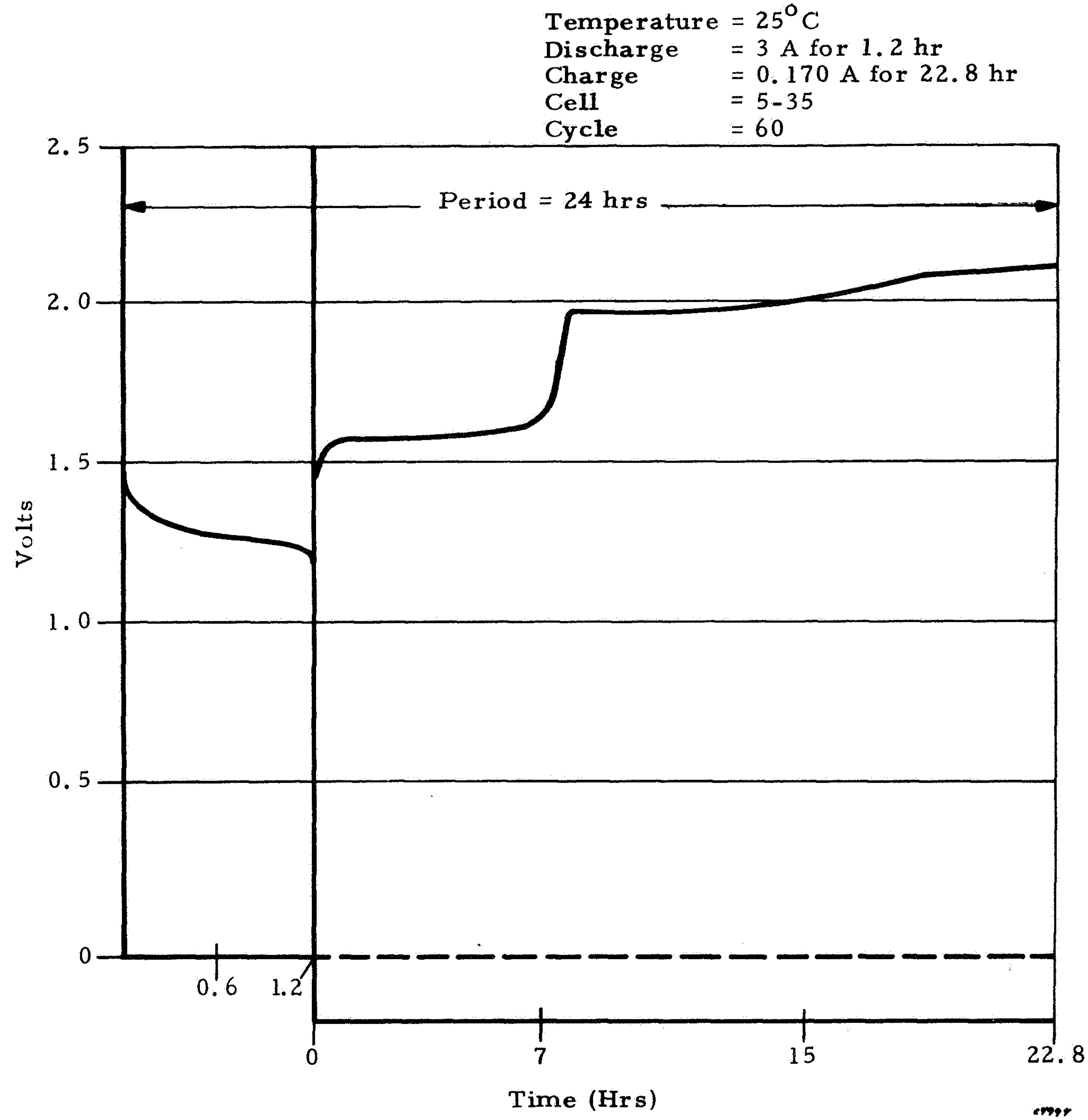


Figure 71. Cycling Curves, Lot #5 - Test C

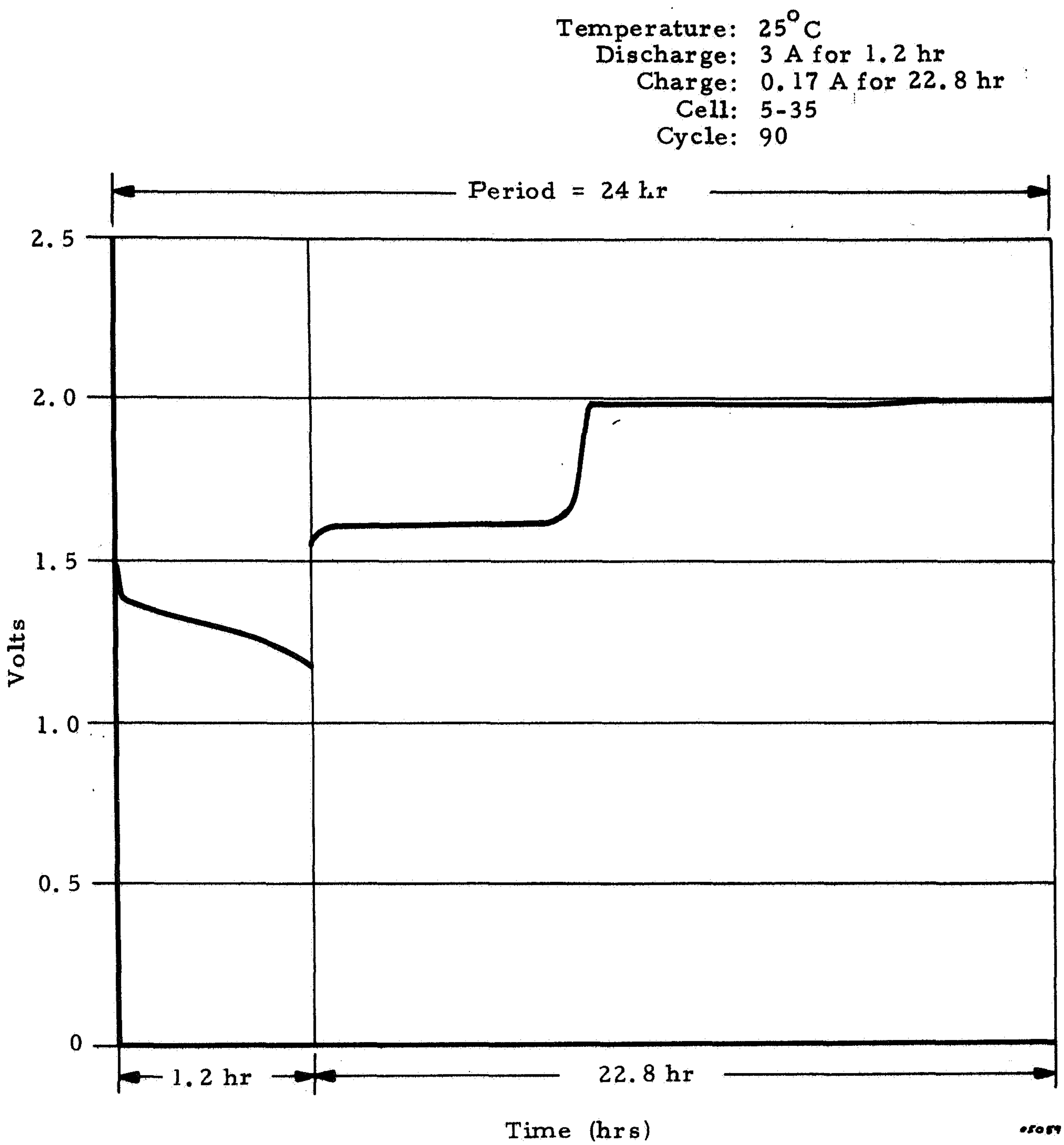


Figure 72. Cycling Curves, Lot #5 - Test C

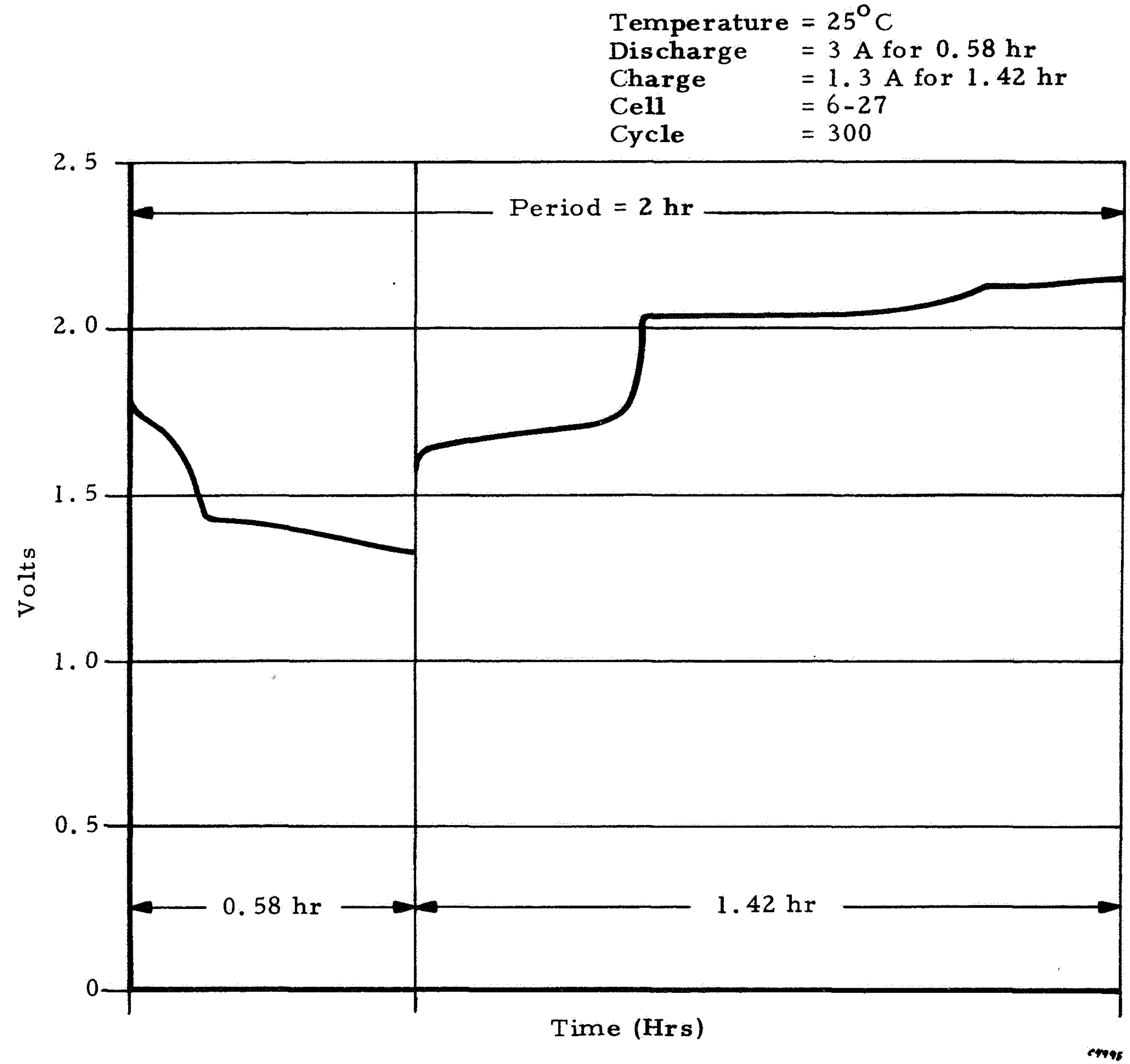


Figure 73. Cycling Curves, Lot #6 - Test D

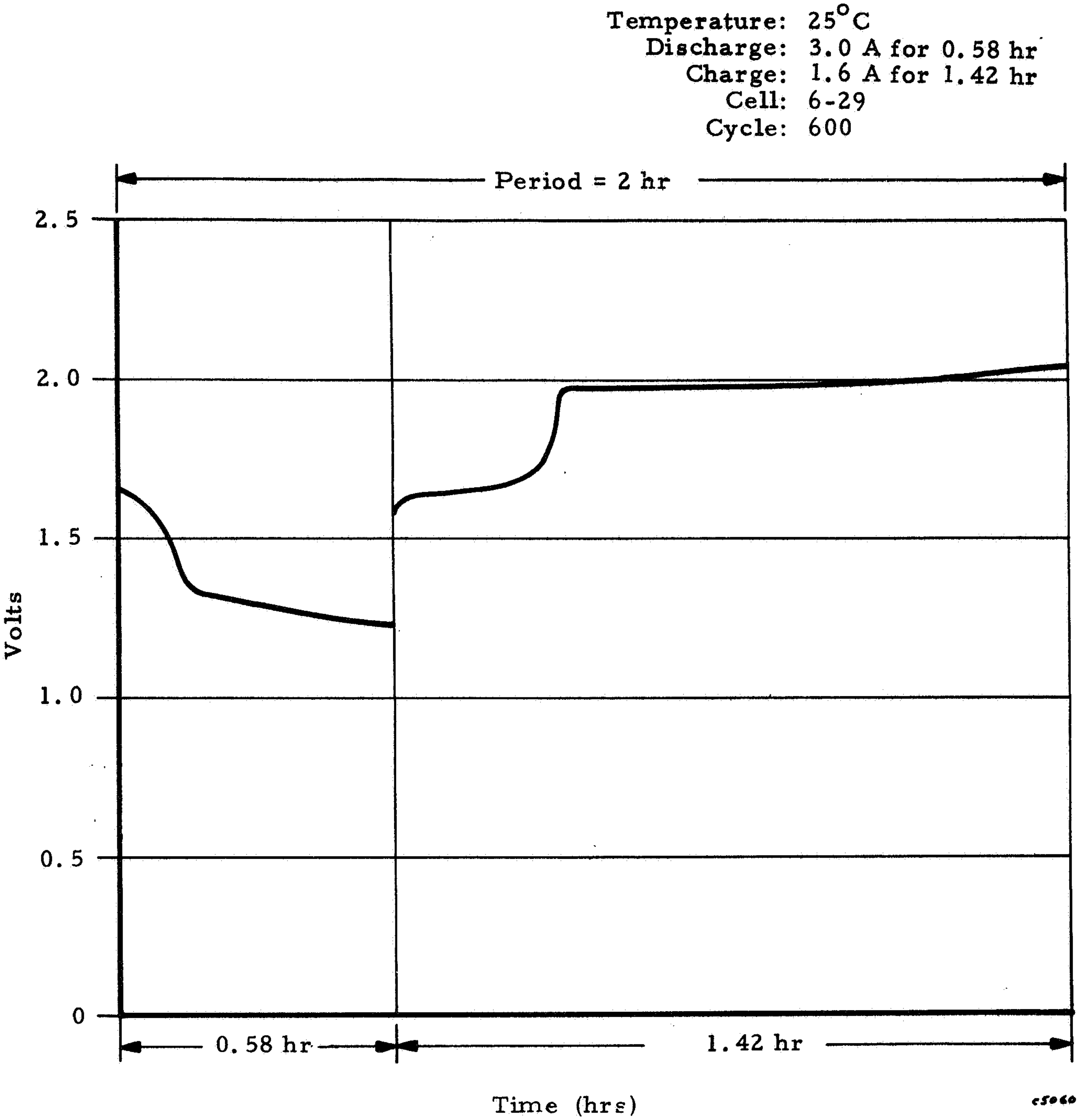


Figure 74. Cycling Curves, Lot #6 - Test D

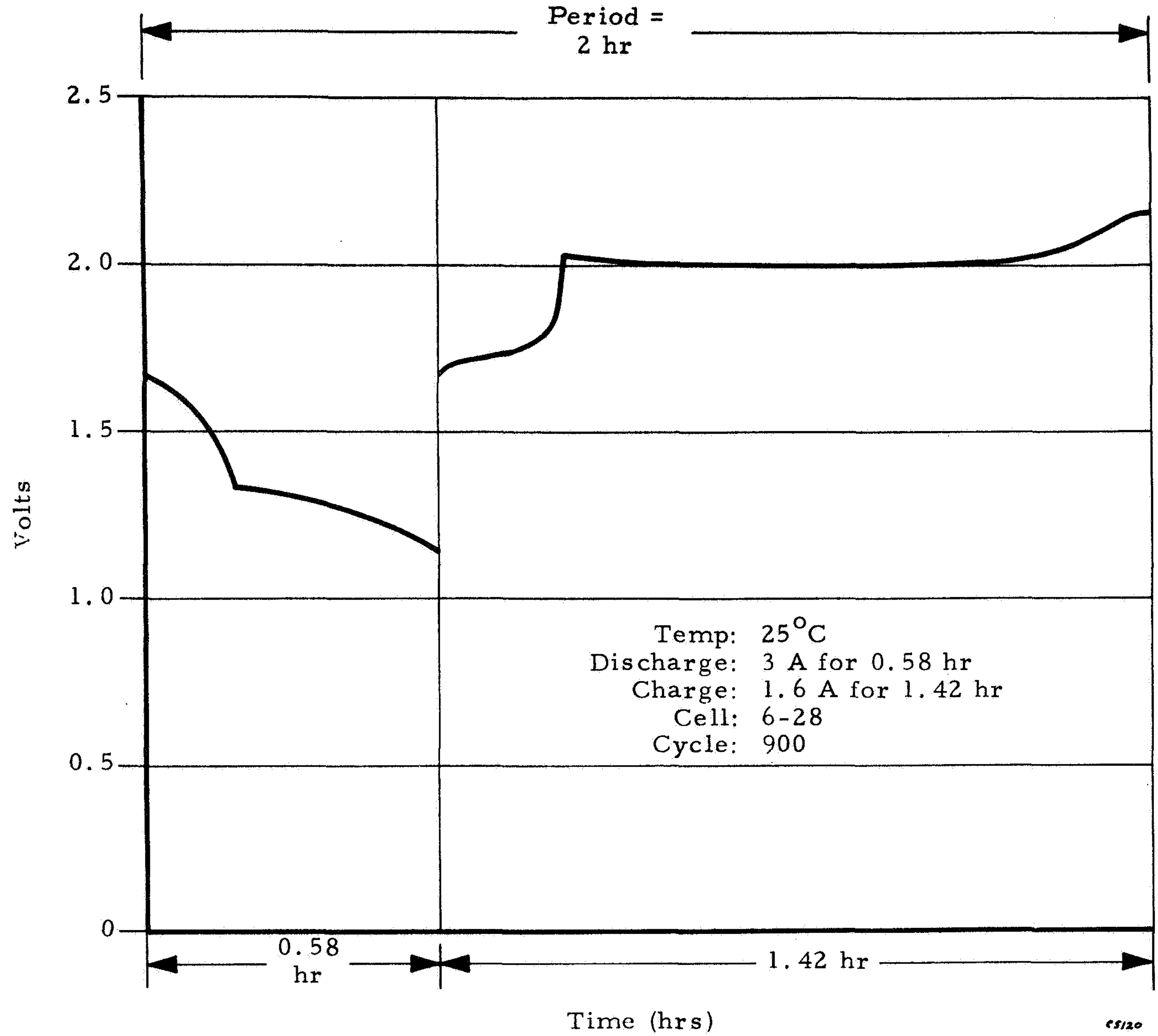


Figure 75. Cycling Curves, Lot #6 - Test D

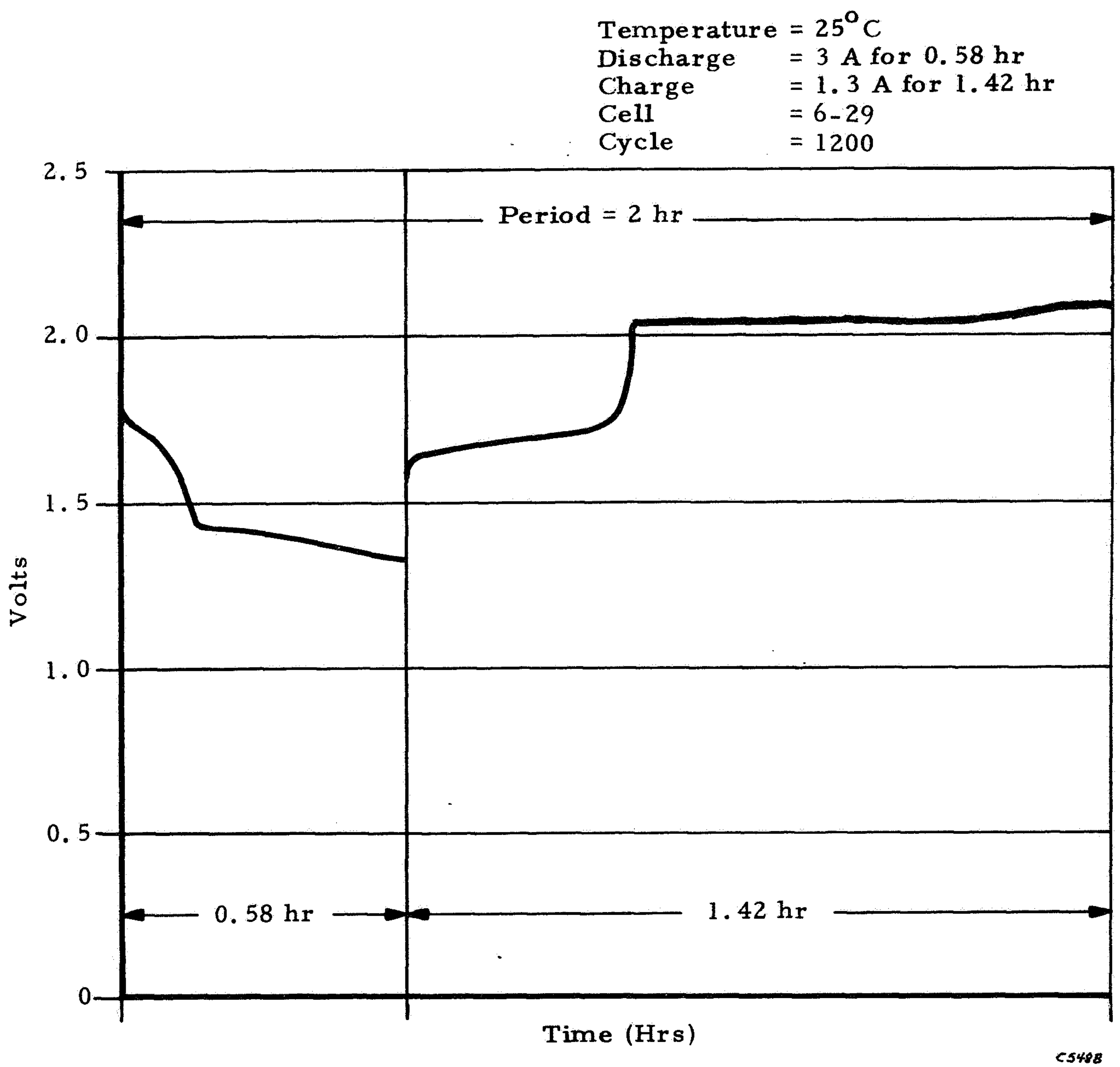


Figure 76. Cycling Curves, Lot #6 - Test D

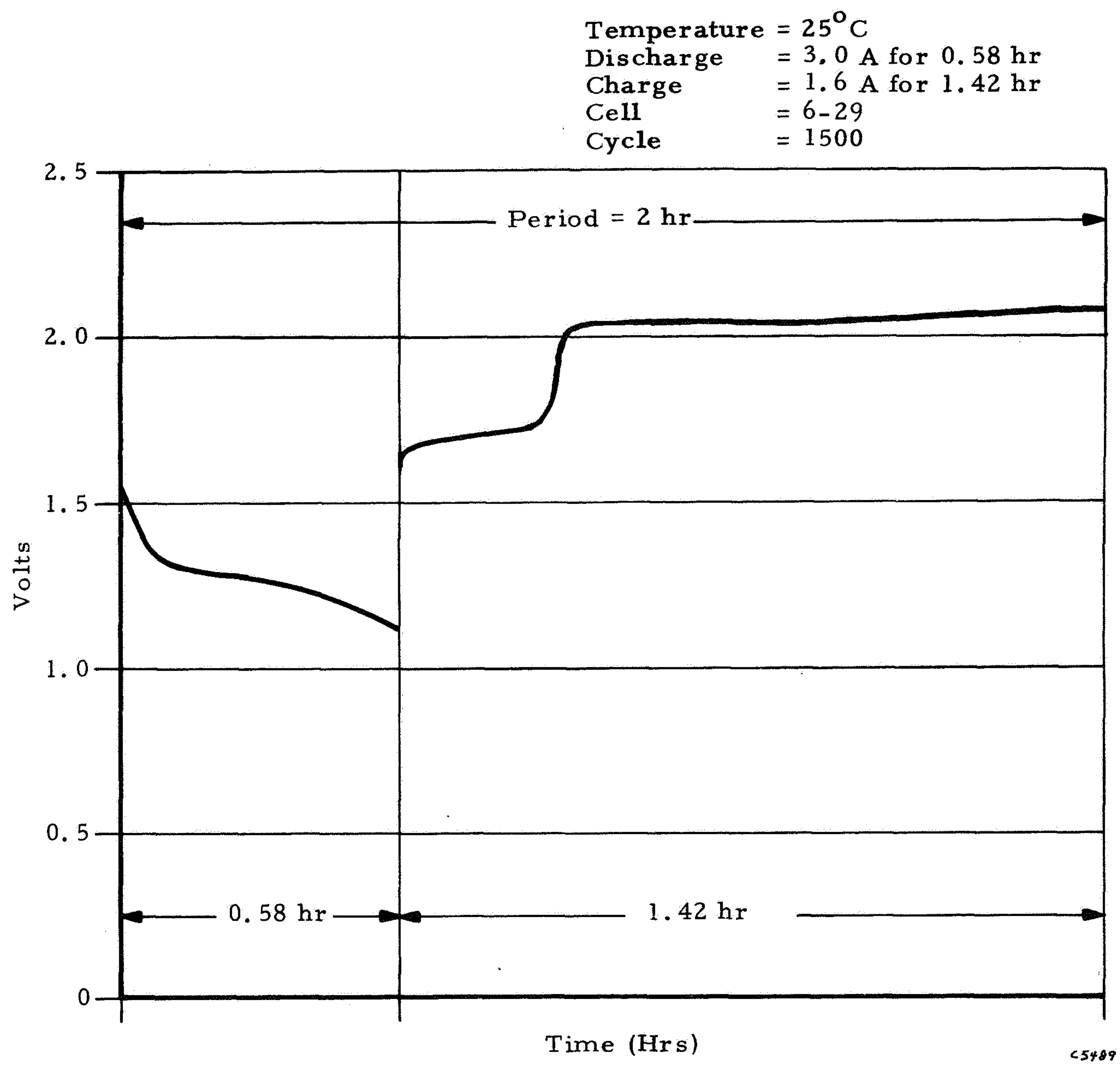


Figure 77. Cycling Curves, Lot #6 - Test E

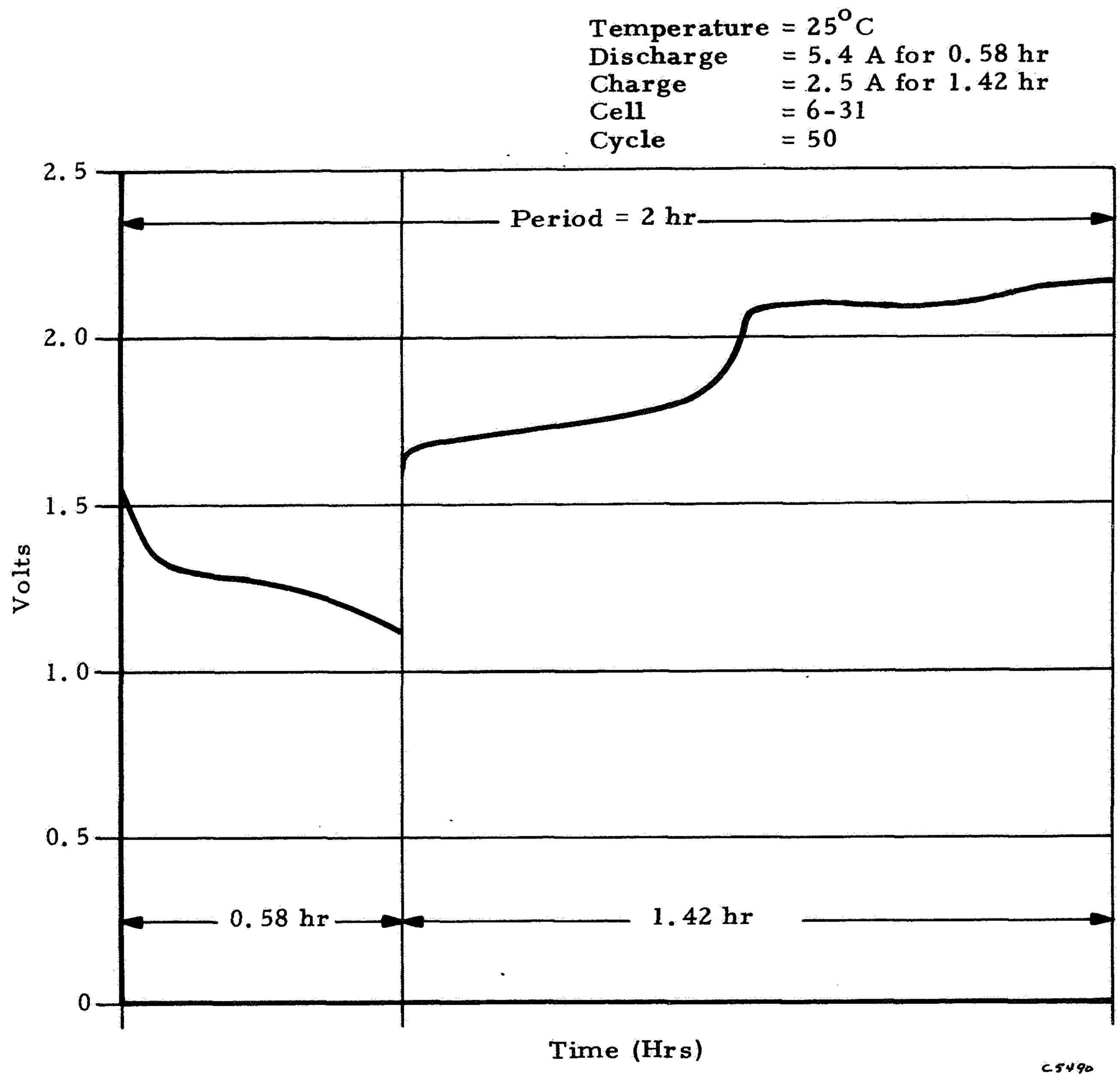


Figure 78. Cycling Curves, Lot #6 - Test E

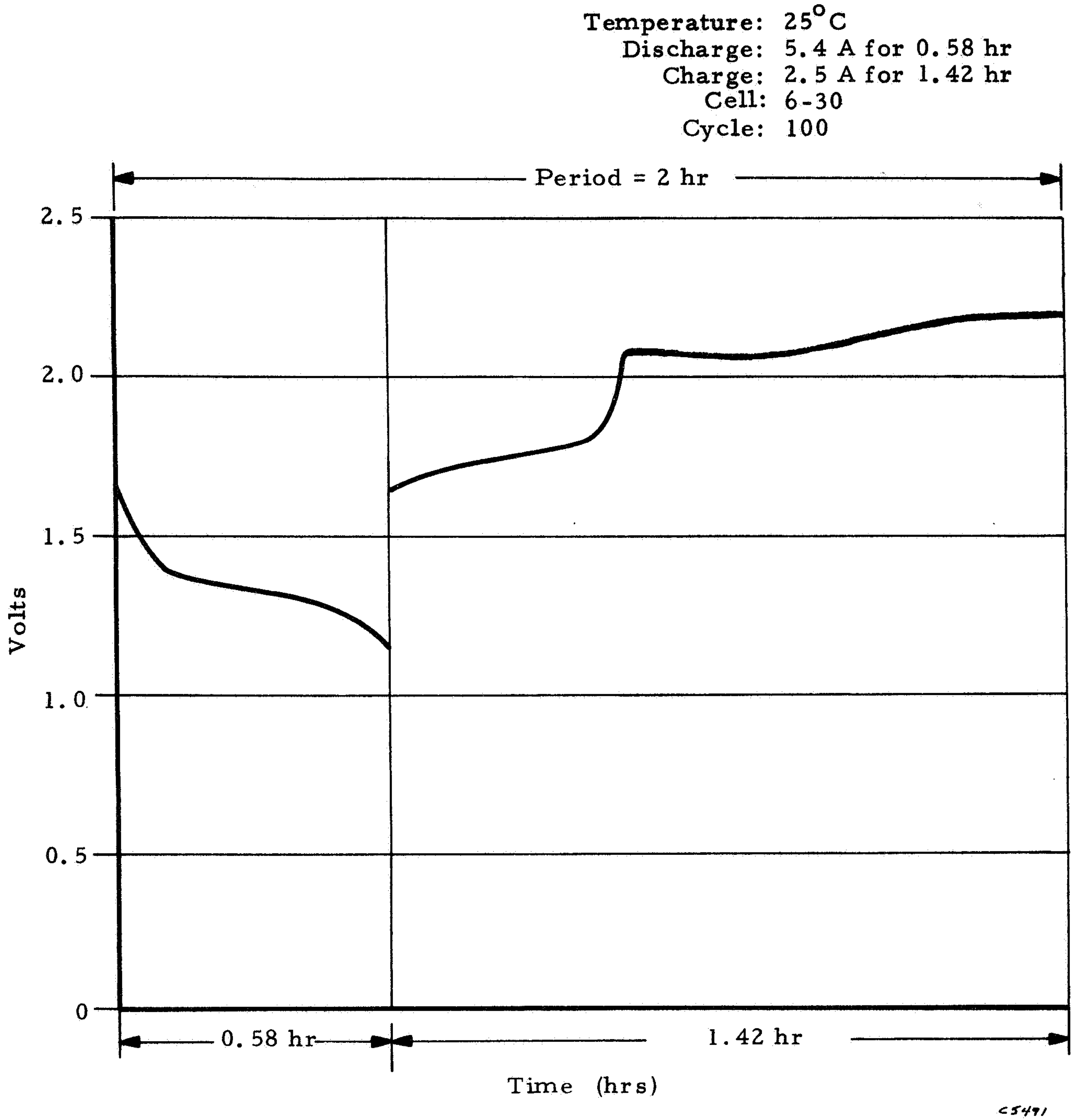


Figure 79. Cycling Curves - Lot #6, Test E

TABLE XC

FIRST CYCLING FAILURES

TEST A

Cycling Failure	Lot 1				Lot 3					Lot 5			
	1-26	1-27	1-28	1-29	3-26	3-27	3-28	3-29	3-36	5-26	5-27	5-28	5-29
1	962	709	978	823	635	448	352	211	377	695	1002	323	1009
2	1045	793	1063	994	847	855	384	243	725	1008	1068	547	1060
3	1255	1036	1278	—	1048	1195	841	769	958	—	1229	1026	1284

TEST B

Cycling Failure	Lot 1				Lot 3					Lot 5			
	1-30	1-31	1-32	1-33	3-30	3-31	3-32	3-39	3-40	5-30	5-31	5-32	5-33
1	326	651	337	324	454	225	458	405	430	283	409	198	646
2	—	—	—	—	—	—	—	—	—	291	—	291	648
3	—	—	—	—	—	—	—	—	—	—	—	—	—

TEST C

Cycling Failure	Lot 1		Lot 3		Lot 5	
	1-34	1-34	3-34	3-35	5-34	5-35
1	51	158	73	86	53	99
2	—	—	—	96	59	101
3	—	—	—	97	64	—

TABLE XCI

FIRST CYCLING FAILURES

TEST D

Cycling Failure	Lot 2				Lot 4				Lot 6			
	2-26	2-27	2-28	2-29	4-26	4-27	4-28	4-29	6-26	6-27	6-28	6-29
1	543	420	531	591	750	350	494	832	878	796	621	1076
2	—	506	598	627	881	352	656	945	911	967	789	1144
3	—	—	731	709	1013	893	805	1051	983	1018	1028	1223

TEST E

Cycling Failure	Lot 2											Lot 4				Lot 6			
	Group I				Group II				Group III										
	2-30	2-31	2-32	2-33	12-21	12-22	12-23	12-24	2-36	2-37	2-38	4-30	4-31	4-32	4-33	6-30	6-31	6-32	6-33
1	55	55	112	30	147	146	198	208	243	254	338	190	334	157	156	92	80	60	55
2	—	—	115	95	158	157	209	258	—	—	352	256	351	162	162	106	115	97	64
3	—	—	126	113	—	371	359	281	—	—	—	259	—	179	—	147	156	167	77

TABLE XCII

LOT #1 ELECTROLYTE ADDITION (cm^3)

(Total amount added between indicated cycles)

Test A					Test B					Test C		
	Cell Number					Cell Number					Cell Number	
Cycles	26	27	28	29	Cycles	30	31	32	33	Cycles	34	35
	0	0	0	0		16	16	17	12		0	0
300					200					30		
	0	0	0	0		15	23	16	14		0	0
600					400					60		
	0	0	0	0			17					0
900					600					90		
	9.5	5	8.5	5			5					0
1200										120		
	4.0		1.0									0
1500												
Total Cycles	2002	895	1493	997		328	651	339	325		51	159
Total Amt. Before Failure	23.5	5	14.5	5		31	61	33	26		0	0
Average per 100 Cycles over Total Life	1.17	0.55	0.97	0.50		9.5	9.4	9.7	8.0		0	0

TABLE XCIII

LOT #2 ELECTROLYTE ADDITION (cm^3)

(Total amount added between indicated cycles)

Cycles	Test D Cell Number				Cycles	Cell Number					
	2-26	2-27	2-28	2-29		2-30	2-31	2-32	2-33	2-38*	12-23*
	0	0	0	0		0	0	0	4	0	0
300					50						
	0	0	2	6		0	0	0	3	0	0
600					100						
			3	6						0	0
900					150						
			4	12						0	0
1200					200						
			6	3						4	33
1500											
Total Cycles	493	453	1783	1233		56	55	126	124	352	461
Total Amt. Before Failure	0	0	26	27		0	0	0	7	4	33
Average per 100 Cycles Over Total Life	0	0	1.45	2.2		0	0	0	5.6	1.15	7.1

*Note: Cell 2-28: Vented, 40% KOH
Cell 12-23: Vented, 30% KOH

TABLE XCIV

LOT #3 ELECTROLYTE ADDITION (cm^3)

(Total amount added between indicated cycles)

Test A					Test B					Test C		
Cycles	Cell Number				Cycles	Cell Number				Cycles	Cell Number	
	26	27	28	29		30	31	32	33		34	35
	0	0	0	0		24	20	25	13		0	0
300					200					30		
	0	0	0	0		22	2	26			0	0
600					400					60		
	12	3	1.5	4		10		9			0	0
900					600					90		
	6	7	7									0
1200										120		
			5									
1500												
Total Cycles	1388	1548	1570	843		456	225	460	112		74	100
Total Amt. Before Failure	18	10	18	9		56	22	60	13		0	0
Average per 100 Cycles over Total Life	1.3	0.65	1.15	0.93		12	10	13	11.5		0	0

TABLE XCV

LOT #4 ELECTROLYTE ADDITION (cm^3)

(Total amount added between indicated cycles)

	Test D					Test E			
	Cell Number					Cell Number			
Cycles	26	27	28	29	Cycles	30	31	32	33
	0	2	3	0		0	0	0	0
300					50				
	6	0	1	0		0	0	0	0
600					100				
	4	5	9	5		0	0	0	0
900					150				
	10	13	6	12		2	0	9	4
1200					200				
				5		10	14		
1500									
Total Cycles	1117	1177	1072	1218		276	351	229	164
Total Amt. Before Failure	20	20	19	22		12	14	11	4
Average per 100 Cycles Over Total Life	1.8	1.7	1.75	1.8		4.4	4.0	4.8	2.45

TABLE XCVI

LOT #5 ELECTROLYTE ADDITION (cm^3)

(Total amount added between indicated cycles)

Test A					Test B					Test C		
	Cell Number					Cell Number					Cell Number	
Cycles	26	27	28	29	Cycles	30	31	32	33	Cycles	34	35
	0	0	0	0		63	44	38	73		0	0
300					200					30		
	0	0	0	0		67	156	53	145		0	0
600					400					60		
	3	3	0	0					145		0	0
900					600					90		
	0	9	4	7					37			0
1200										120		
	0	5	0	4								
1500												
Total Cycles	1025	1356	1604	1401		291	409	290	648		64	101
Total Amt. Before Failure	3	17	6	11		130	200	91	400		0	0
Average per 100 Cycles over Total Life	0.30	1.25	0.37	0.86		4.5	4.9	3.1	6.2		0	0

TABLE XCVII

LOT #6 ELECTROLYTE ADDITION (cm^3)

(Total amount added between indicated cycles)

Test D					Test E				
	Cell Number					Cell Number			
Cycles	26	27	28	29	Cycles	30	31	32	33
	1	1	1	1		0	0	0	0
300					50				
	1	0	0	0		0	0	1	0
600					100				
	18	3	2	4		6	0		
900					150				
	4	4	4	7		4			
1200					200				
			1	3					
1500									
Total Cycles	983	1028	1298	1570		157	156	167	77
Total Amt. Before Failure	24	8	8	18		10	0	1	3
Average per 100 Cycles Over Total Life	2.5	0.78	0.62	1.15		6.4	0	0.6	3.9

attempted in graphical form within the limitations set by the test conditions: very few cells tested on one particular variation and only two points obtained to draw curves (except cycles vs depth of discharge). However, it seemed reasonable from previous experience that data would fit a semi-logarithmic curve.

Figure 80 shows the number of cycles vs depth of discharge.

Figure 81 shows the number of cycles vs current density or current.

Figure 82 shows the number of cycles vs temperature.

One probability curve is shown in Figure 83. Using data of Test A (1.5 hr-period, 3 A discharge, 25°C) and excluding extreme results (at both ends) as having a low probability of occurrence, the curve is plotted giving the probability of exceeding a given number of cycles N as a function of N. It appears relatively linear on a probability scale paper showing a random distribution of the data with no major interference. For instance, the probability of exceeding 1000 cycles is 90% and that of exceeding 1400 cycles only 50%.

Note: All cells of the lot #6 were made with negative electrodes incorporating 1% PbO additive in the zinc oxide mix as requested by the NASA Project Monitor. Overall, the data were not different from the other cells, considering the wide scatter. However, note should be made of the fact that the first cycling failure of the cells of this lot on test D occurred later than in cells of other lots placed on the same test. Cells of lots #6 had about 800 cycles on the first failure, whereas cells of lots #2 and #4 had 500 cycles on the average (see Table XCI).

3.4 OTHER WORK

To determine whether the cell components on the cell fabrication have changed, it was deemed necessary, in agreement with the NASA Program Manager, to run a series of identical tests on 15 cells using design variations inspired from the few changes made during the course of the program and which may have been conducive to the disparity in performance observed on the lots of Task III.

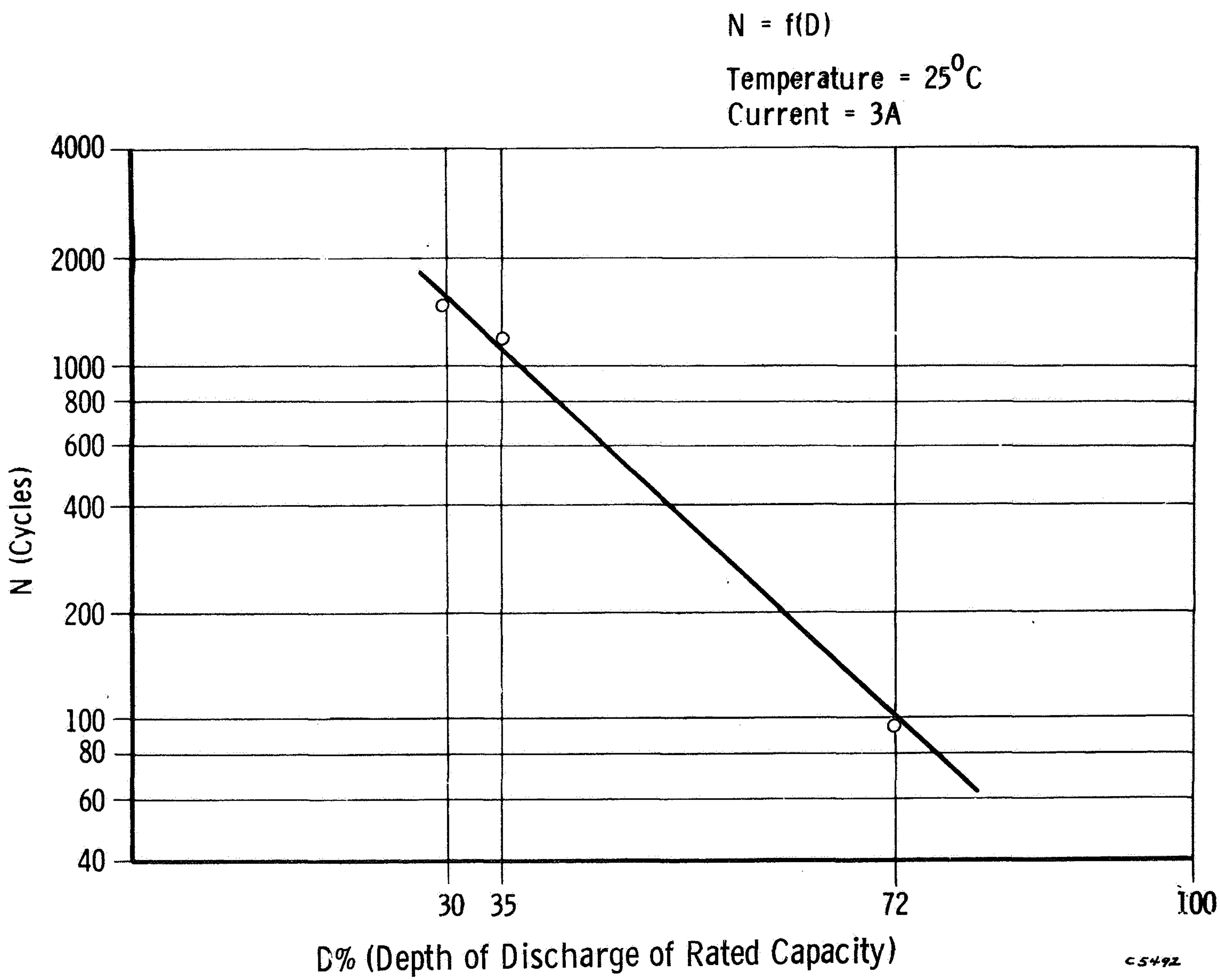


Figure 80. Number of Cycles vs Depth of Discharge

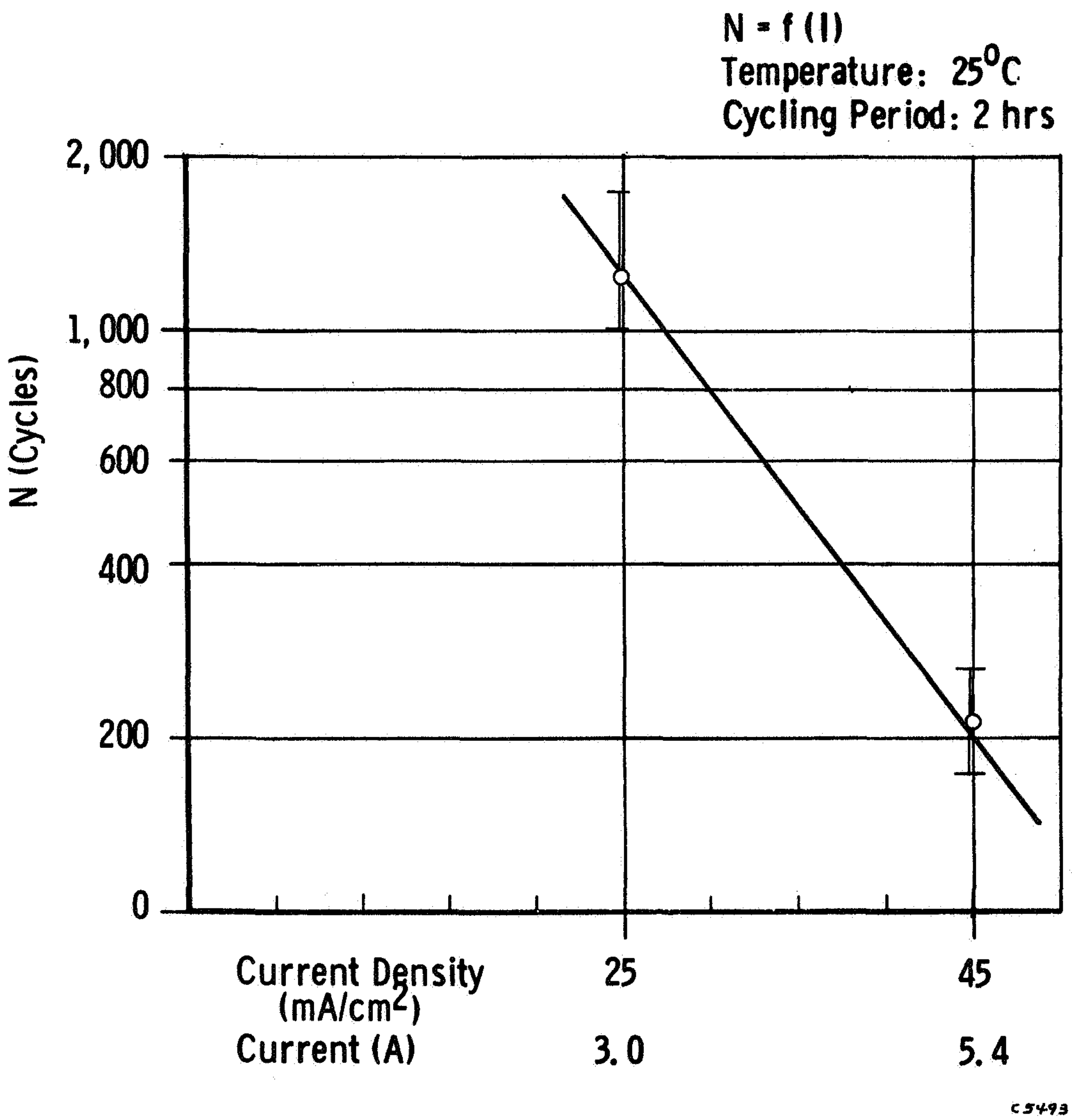


Figure 81. Number of Cycles vs Current Density

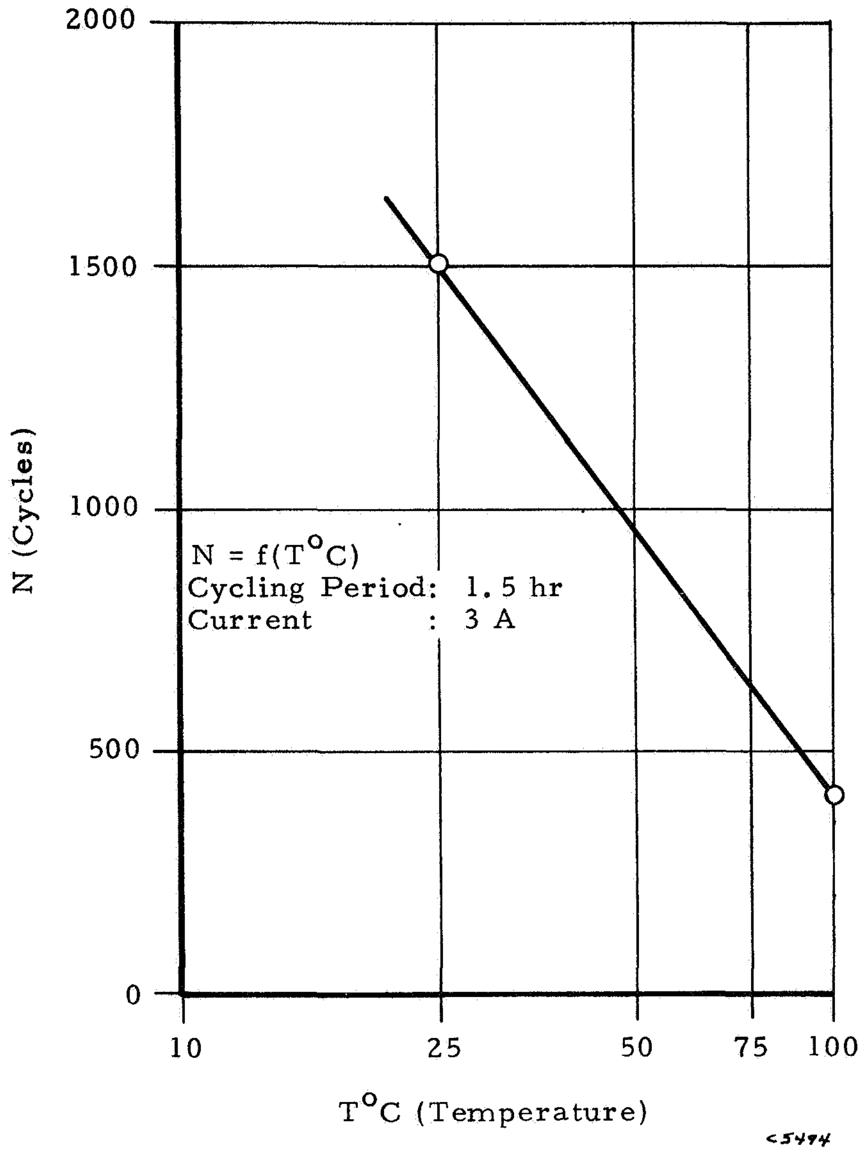


Figure 82. Number of Cycles vs Temperature

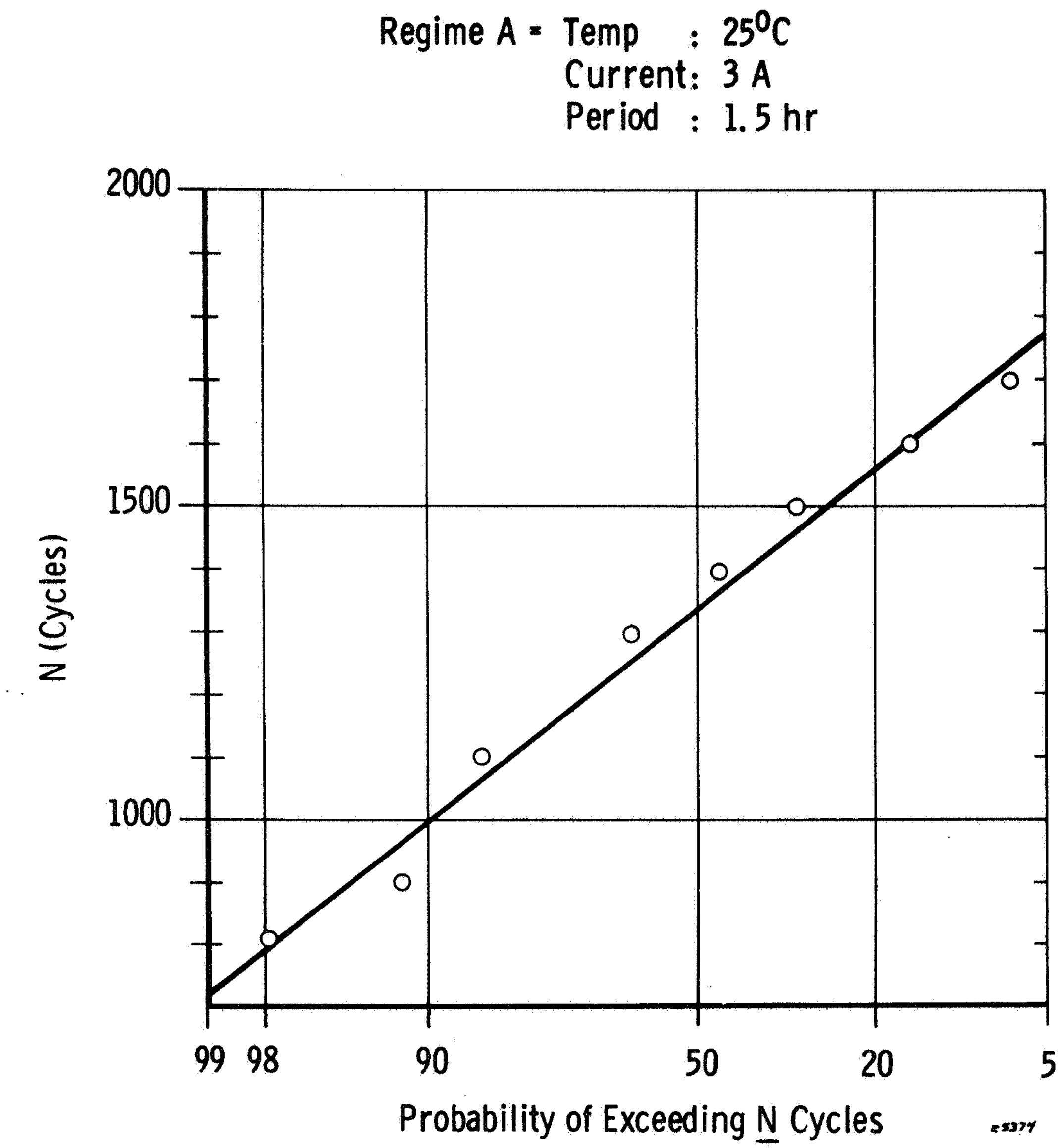


Figure 83. Statistical Distribution

The possible factors may have been:

1. Pellon wetting agent (reputed undesirable)
2. Separator treatment
3. KOH concentration change from 30 to 40%.

Another factor to remember is the cycling method; the cycling was previously done on an individual cell basis rather than on series-connected cells as was done in the beginning of Task III. Therefore, the cells of the present series are being cycled on the same regime used in the previous contract and in the beginning of this program, namely:

1. 90-minute cycling period
2. 2.5 A discharge rate for 1/2 hour
3. 2.1 V voltage limit set from the beginning of cycling
4. Individual cell cycling.

The variations considered in cells of the present series are the following (3 cells per group):

Group A	Regular Pellon taken from the same lot used in all cells over the past two years; this group is actually a control group; that is the present design (electrolyte 40% KOH).
Group B	Pellon washed over 72 hours in distilled water to eliminate or reduce the amount of wetting agent, then dried (electrolyte 40% KOH).
Group C	KT in place of Pellon (electrolyte 40% KOH).
Group D	Same as Group A except the original 3420-09 separator is used in place of the 3420-09 coated with a 1 mil layer of 3420-25; this treatment was introduced at the end of Task II to minimize gassing (electrolyte 40% KOH).
Group E	Same as Group D except 30% KOH is used; this type of cell is actually the original cell design used at the beginning of the program.

Table XCVIII gives the original electrical characteristics of the 15 cells (capacity and plateau voltage). Table IC gives their cycling data. Table C gives the number of maintenances done on each cell after each cycling failure, until the cell was deemed unsuitable for further cycling or had a catastrophic

TABLE XCVIII

FORMATION CAPACITY OF CELLS OF VARIOUS CONSTRUCTION FEATURES

Variation*	Cell No.	Output (Ah)	Plateau Voltage (V)
A (Present Design)	ZL-60-1	6.85	1.46
	ZL-60-2	6.95	1.44
	ZL-60-3	6.90	1.45
	Average:	6.90 Ah	1.45 V
B	ZL-60-4	7.25	1.45
	ZL-60-5	7.25	1.45
	ZL-60-6	7.35	1.45
	Average:	7.30 Ah	1.45 V
C	ZL-60-7	6.95	1.45
	ZL-60-8	6.65	1.45
	ZL-60-9	6.60	1.45
	Average:	6.75 Ah	1.45 V
D	ZL-60-10	6.45	1.44
	ZL-60-11	6.80	1.44
	ZL-60-12	6.80	1.43
	Average:	6.70 Ah	1.44 V
E (Original Design)	ZL-60-13	7.05	1.45
	ZL-60-14	6.85	1.43
	ZL-60-15	6.90	1.44
	Average:	6.95 Ah	1.44 V

(*See details in report)

Formation

Charge: 0.350 A to 2.05 V

Discharge: 1 A to 1.0 V

TABLE IC

VARIOUS CONSTRUCTION FEATURES CYCLING DATA

Regime:

Discharge: 2.5 A for 1/2 hr.
Charge: 1.5 A for 1 hr.
Individual cell cycling

Group	Separator	Positive Interseparator	KOH	Cell No.	Cycles
A (present control)	3420-09 coated	Pellon	40%	ZL-60-1	1242
				ZL-60-2	1501
				ZL-60-3	1281
B	3420-09 coated	Washed Pellon	40%	ZL-60-4	1240
				ZL-60-5	1055
				ZL-60-6	1127
C	3420-09 coated	KT	40%	ZL-60-7	1339
				ZL-60-8	1314
				ZL-60-9	1334
D	3420-09	Pellon	40%	ZL-60-10	1115
				ZL-60-11	1157
				ZL-60-12	909
E (original control)	3420-09	Pellon	30%	ZL-60-13	402
				ZL-60-14	937
				ZL-60-15	1066

TABLE C

MAINTENANCE FREQUENCY (CELLS ZL-60)

(Cycle Number at Which Maintenance Was Done)

Group	A			B			C			D			E		
Maintenance \ Cell Number ZL-60	-1	-2	-3	-4	-5	-6	-7	-8	-9	-10	-11	-12	-13	-14	-15
1	705	709	733	749	979	929	1060	1212	1268	583	790	665	160	420	403
2	912	998	991	1098	1054	979	1114	1304	1334	683	919	725	195	725	981
3	950	1263	1052	1223		1115	1284	1314		902	1110	736	347	880	1065
4	1187	1307	1177	1240		1127	1314			1115	1157	842	368	927	
5	1237	1411	1224				1339					897	399	937	
6	1242	1422	1271									909	402		
7		1436	1281												
8		1515													

failure. Only the group C (with KT) exceeded 1000 cycles before having the first cycling failure, and the three cells were remarkably consistent in their total cycle number (1314 to 1339 cycles). Table CI giving the amount of electrolyte addition during the cycle life of all cells, shows again that group C is ahead in performance with only 4 cm^3 used up to 1000 cycles, whereas the control group A used 6 cm^3. Moreover, no electrolyte was added before cycle 800 in group C, whereas the control group A used already 3 cm^3 long before cycle 800.

Upon dissection of the cells, all cells except group C exhibited cracked separators in the usual form (lower part of the wafer) resulting from the slumping and expansion of the zinc electrode. Group C cells were remarkable in the following aspect: zinc electrodes were not slumped and the separator-electrode wafer assemblies were not swollen and appeared flat with only a very fine diagonal crack in one separator.

Without commenting on the merit of KT, it should be pointed out that Pellon was common to all the other cells which appeared to have contributed to the disparity of their cycling performance.

Cycling curves at various cycles are shown for the 5 groups:

Group A	Figures 84 to 87
Group B	Figures 88 to 90
Group C	Figures 91 to 93
Group D	Figures 94 to 95
Group E	Figures 96 to 98

3.5 EFFECT OF CYCLING METHOD

Ten standard cells were tested on the same regime as described in Paragraph 3.4 to determine the effect of series-connection cycling versus individual-connection cycling on the performance of the cells. The 10 cells were divided as follows: five cells were tested in series as a battery on a single panel (however, every time a cell failed, it was removed and the remaining cells continued cycling as a battery) and five cells were placed on individual panels and power supplies. Table CII gives their cycling data.

TABLE CI

ELECTROLYTE ADDITION (cm^3)
TOTAL AMOUNT BETWEEN INDICATED CYCLES
(ZL-60 CELLS)

Group →	A			B			C			D			E		
Cycle / Cell →	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
0 – 200	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
200 – 400	0	0	0	0	0	0	0	0	0	1	1	1	5	5	3
400 – 600	0	0	0	0	0	0	0	0	0	4	1	1	5	3	2
600 – 800	3	3	3	3	0	0	0	0	0	2	4	6		6	3
800 – 1000	4	1	3	4	4	7	4	4	4	2	0	6		5	4
1000 – 1200	4	0	4	3	0	3	3	3	3	3	6			3	
1200 – 1400	0	5	3	2			6	6	2						
1400 – 1600		3													
Cumulative up to 1000 cycles	7	4	6	7	4	7	4	4	4	9	6	14	10	19	12
Average per group up to 1000 cycles	6			6			4			10			14		
Total to Failure	11	12	13	12	4	10	13	13	9	12	12	14	10	22	12
Average per 100 cycles over total life	0.89	0.78	1.0	1.0	0.4	1.05	1.0	1.0	0.68	1.07	1.05	1.5	2.5	2.4	1.1
Average per 100 cycles per group	0.9			0.8			0.9			1.2			2.0		

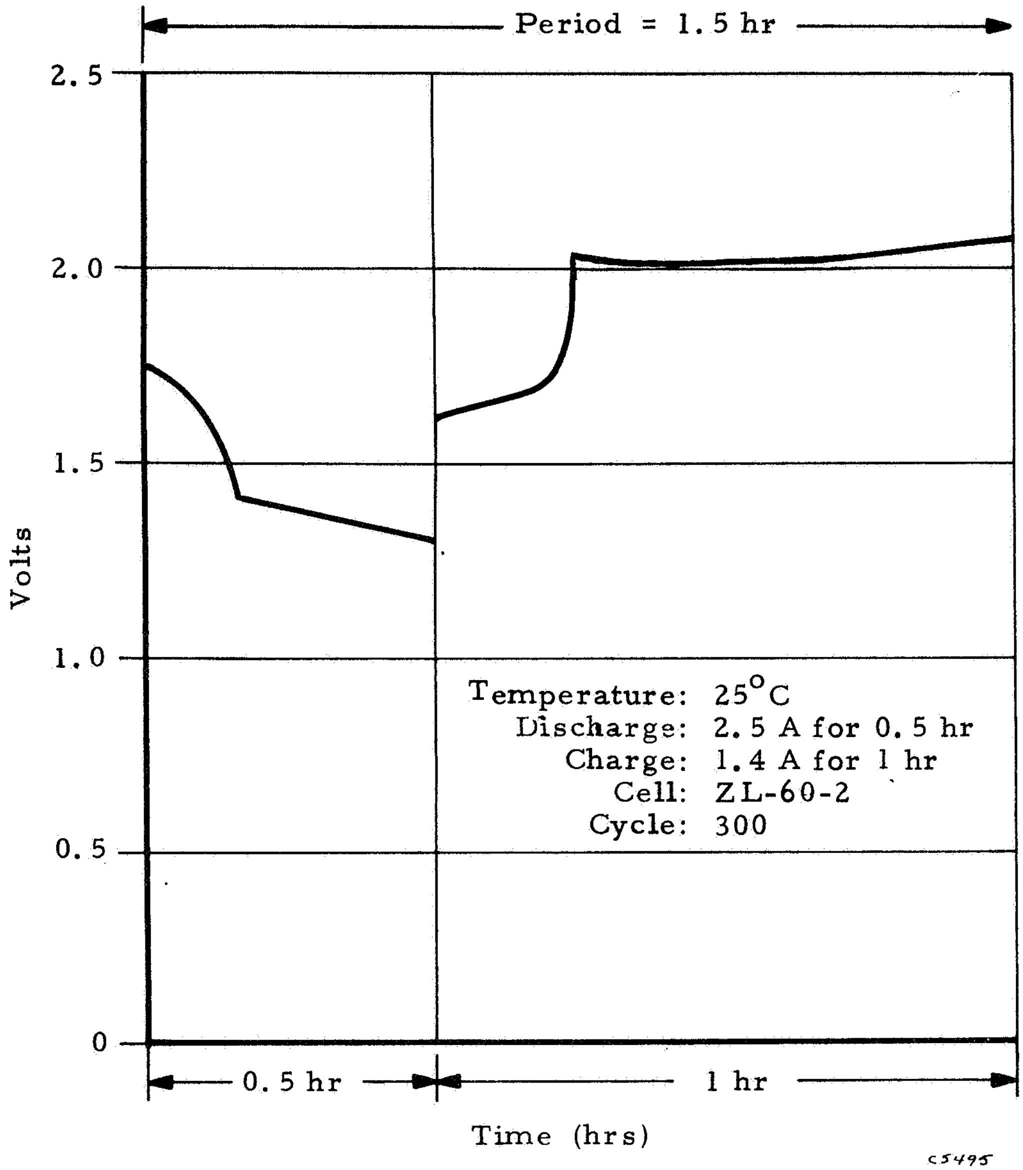


Figure 84. Cycling Curves - Group A

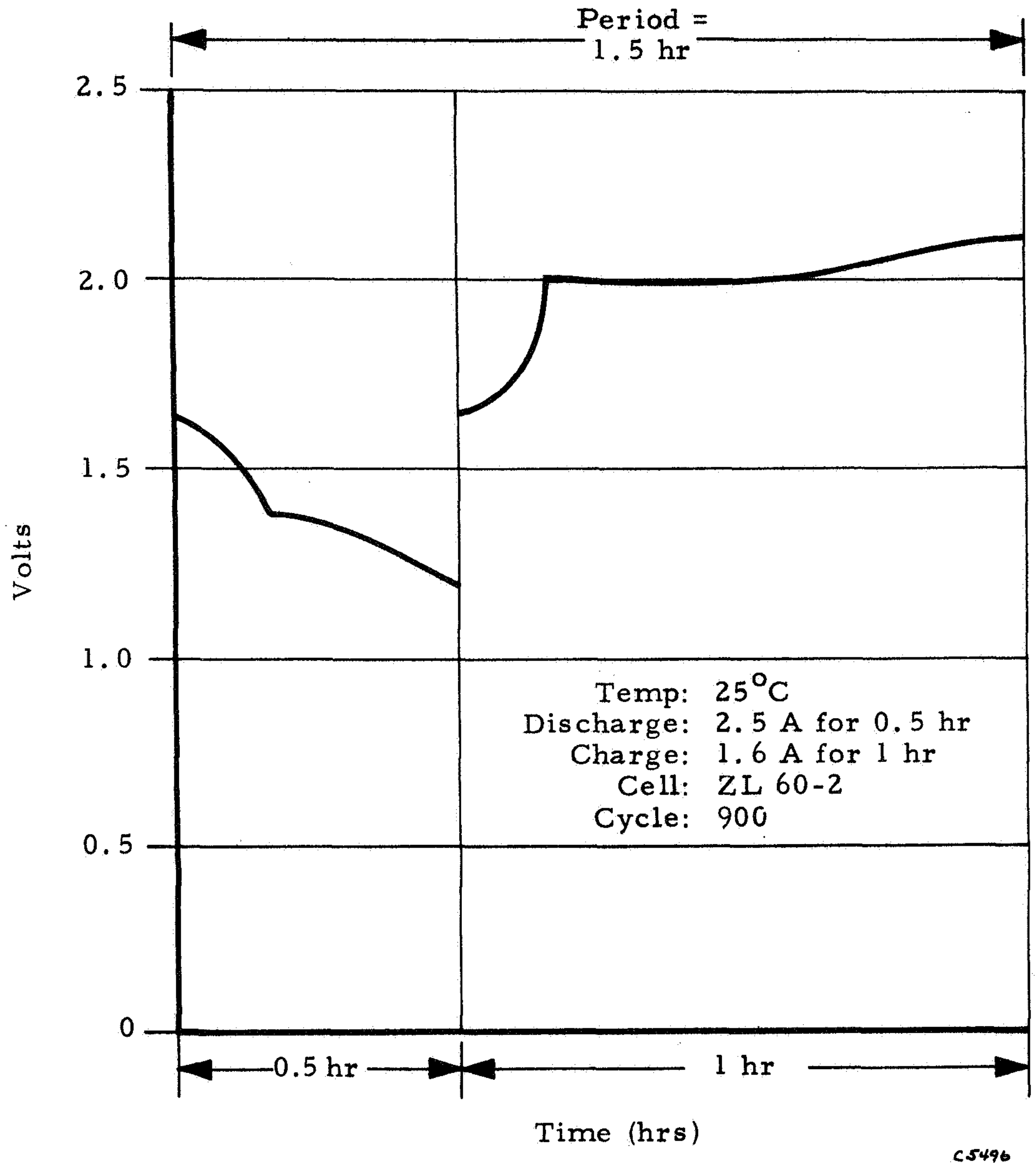


Figure 85. Cycling Curves - Group A

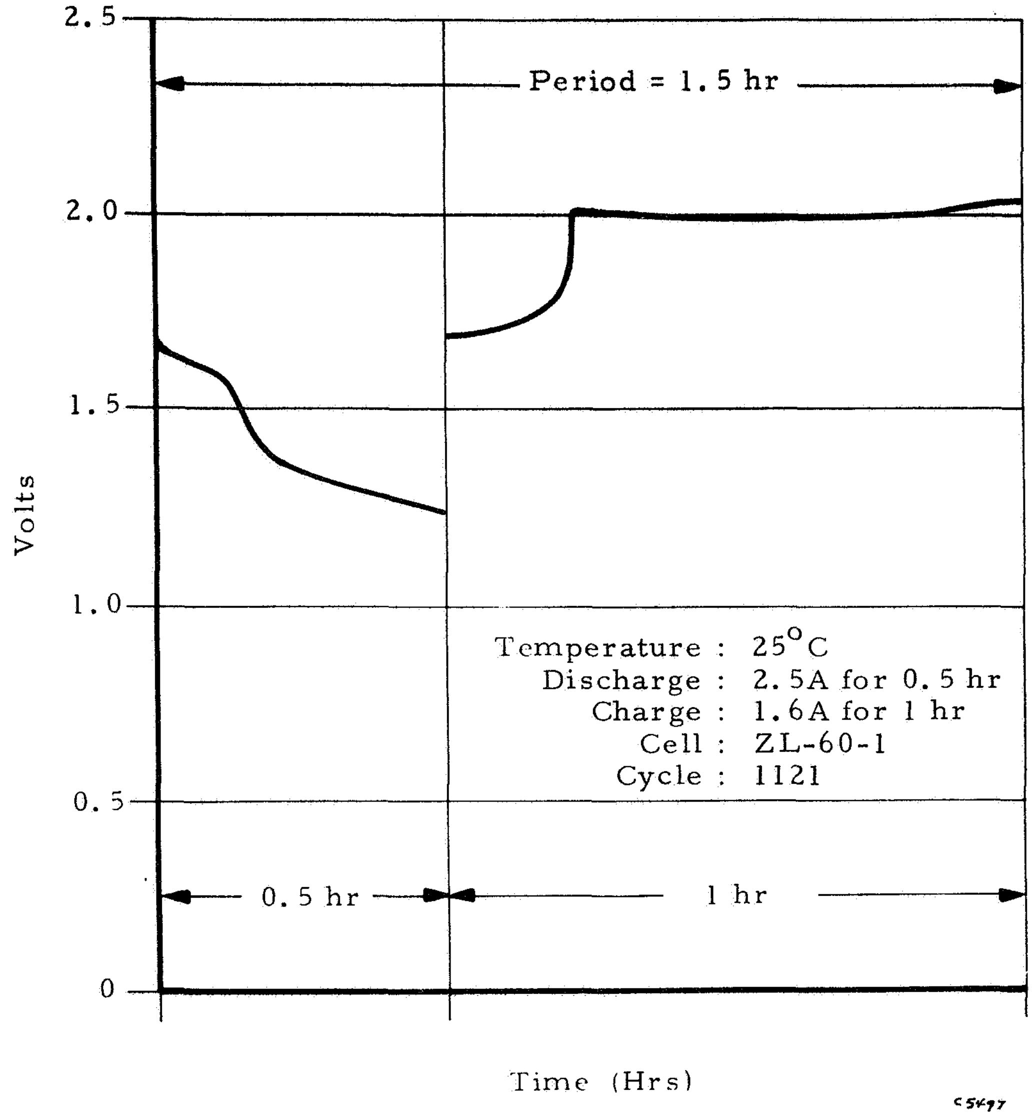


Figure 86. Cycling Curves - Group A

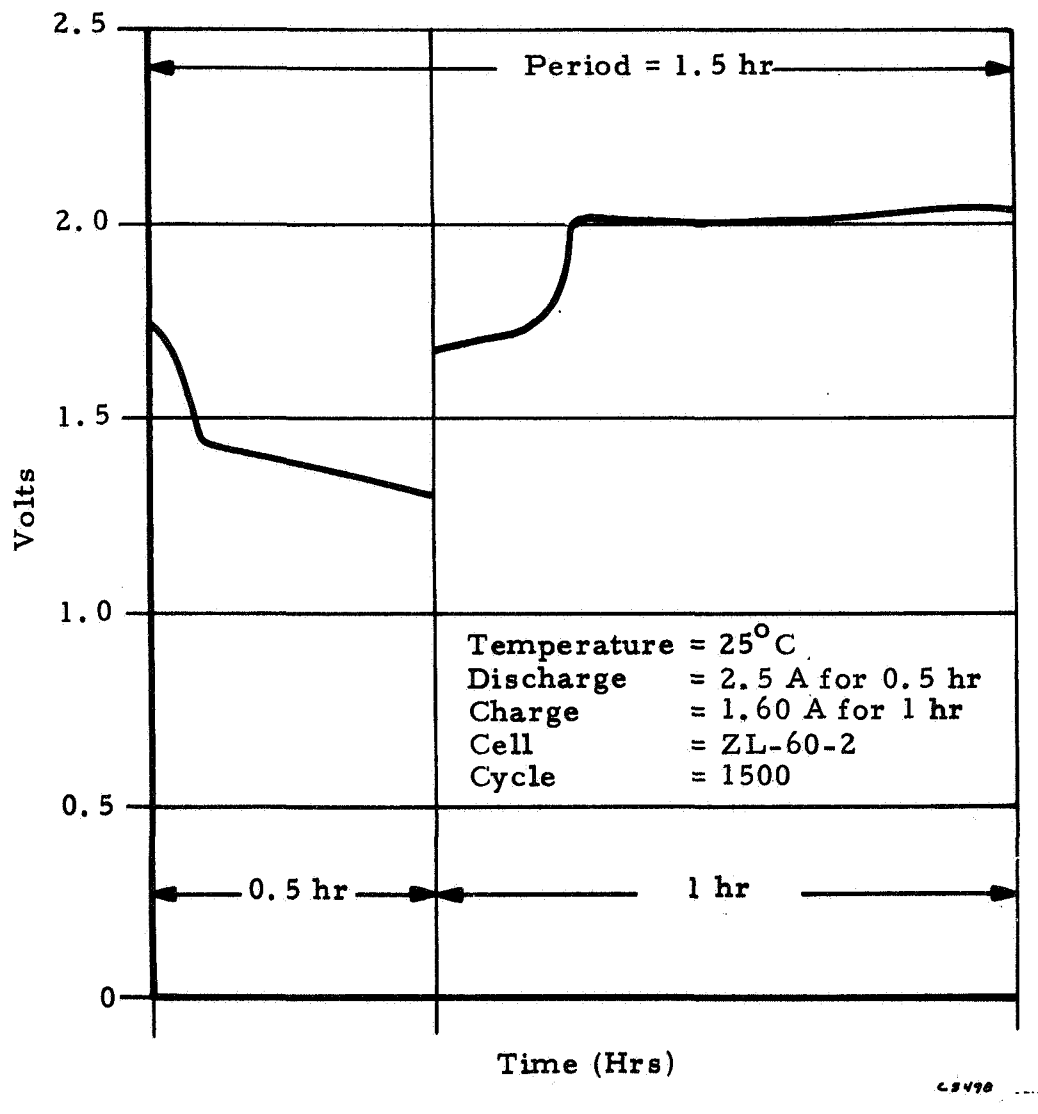


Figure 87. Cycling Curves - Group A

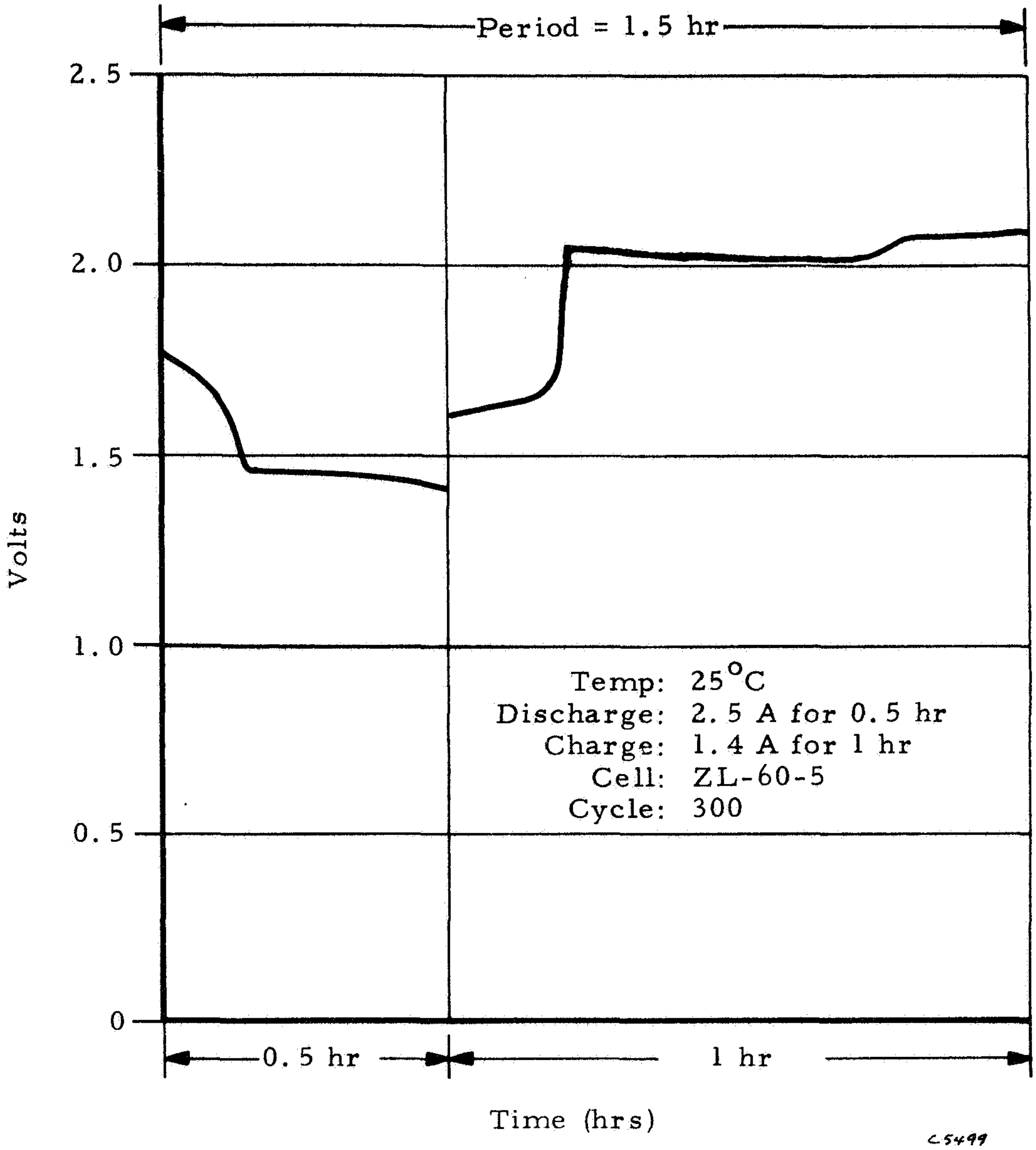


Figure 88. Cycling Curves - Group B

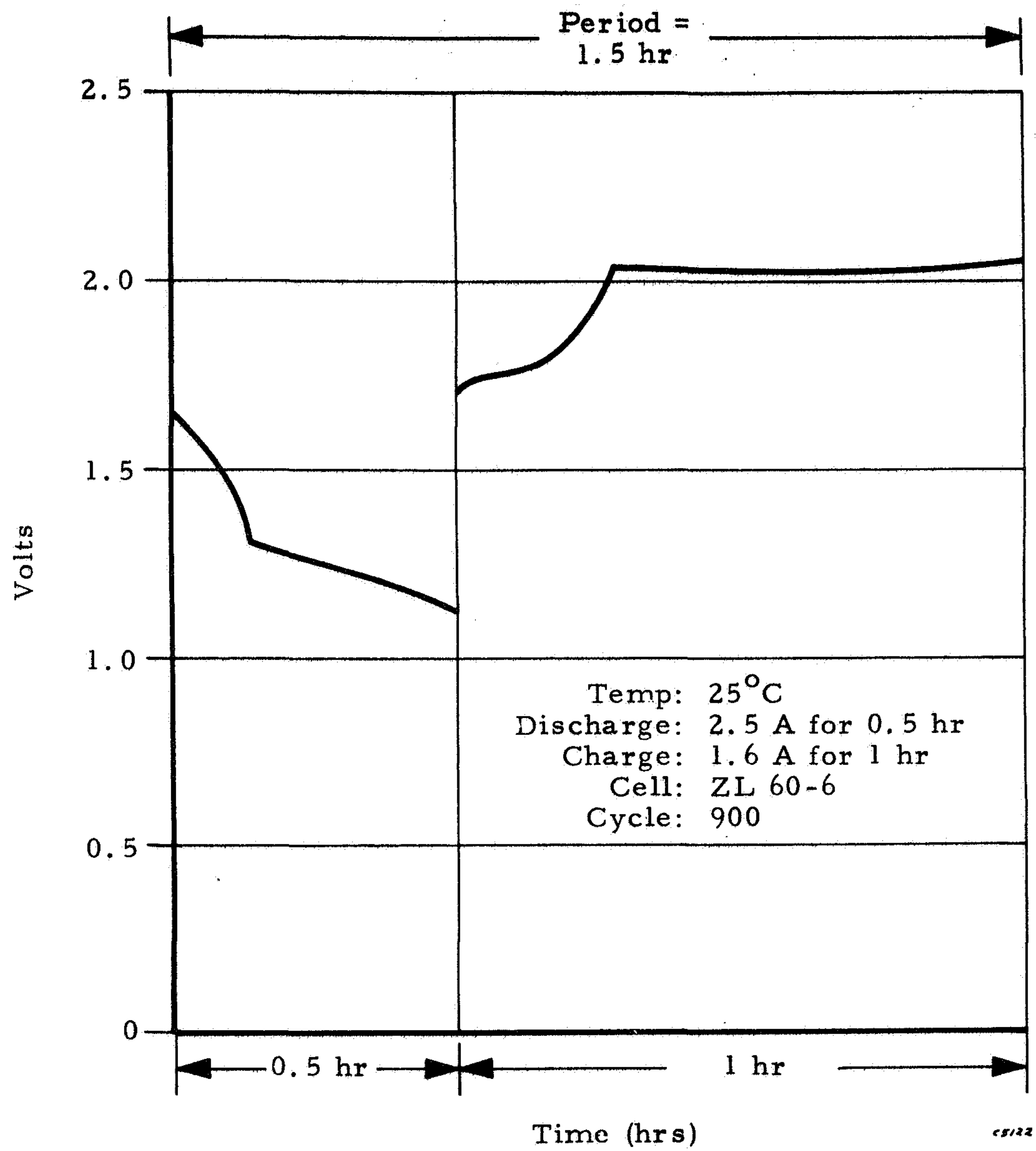


Figure 89. Cycling Curves - Group B

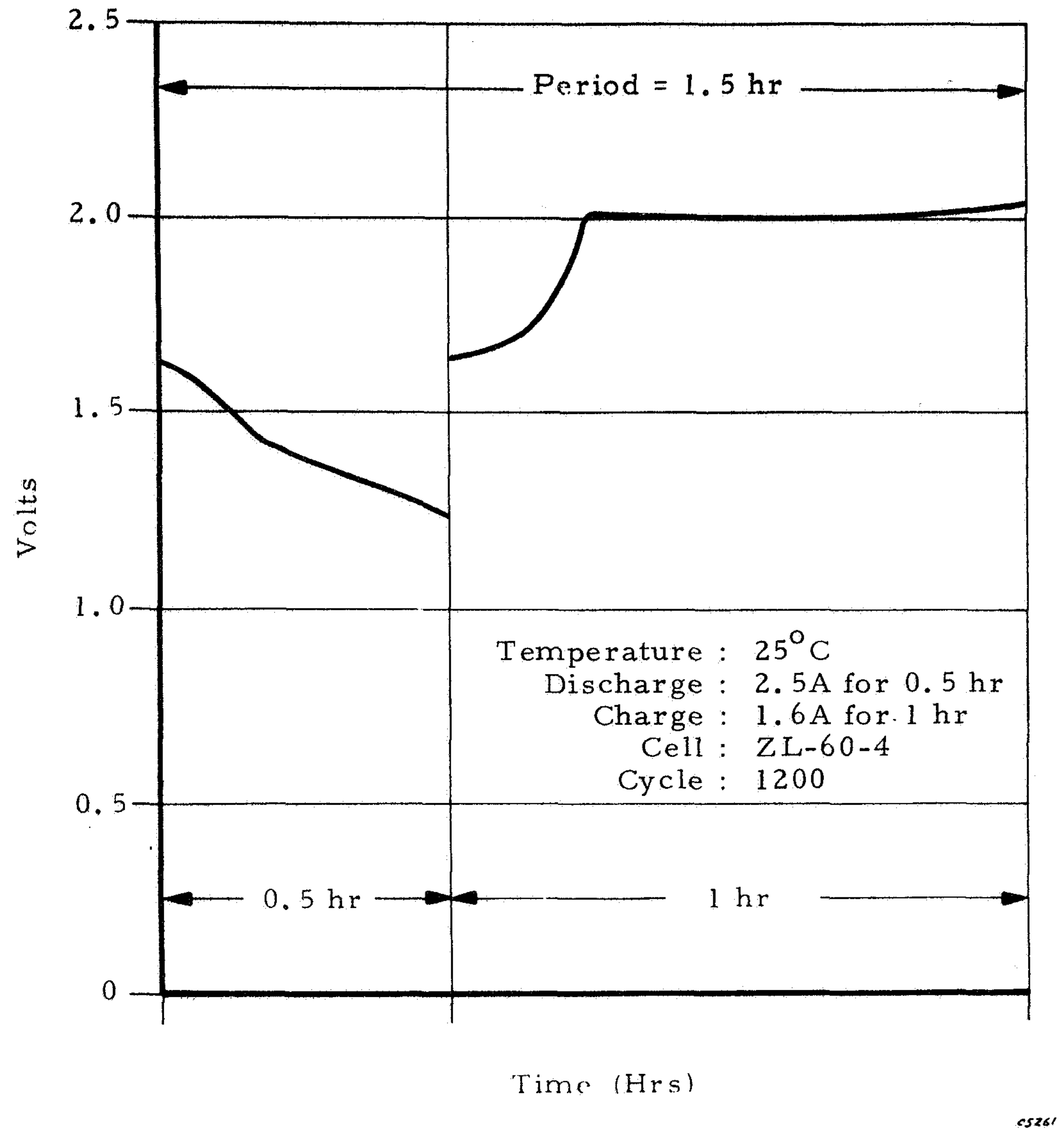


Figure 90. Cycling Curves - Group B

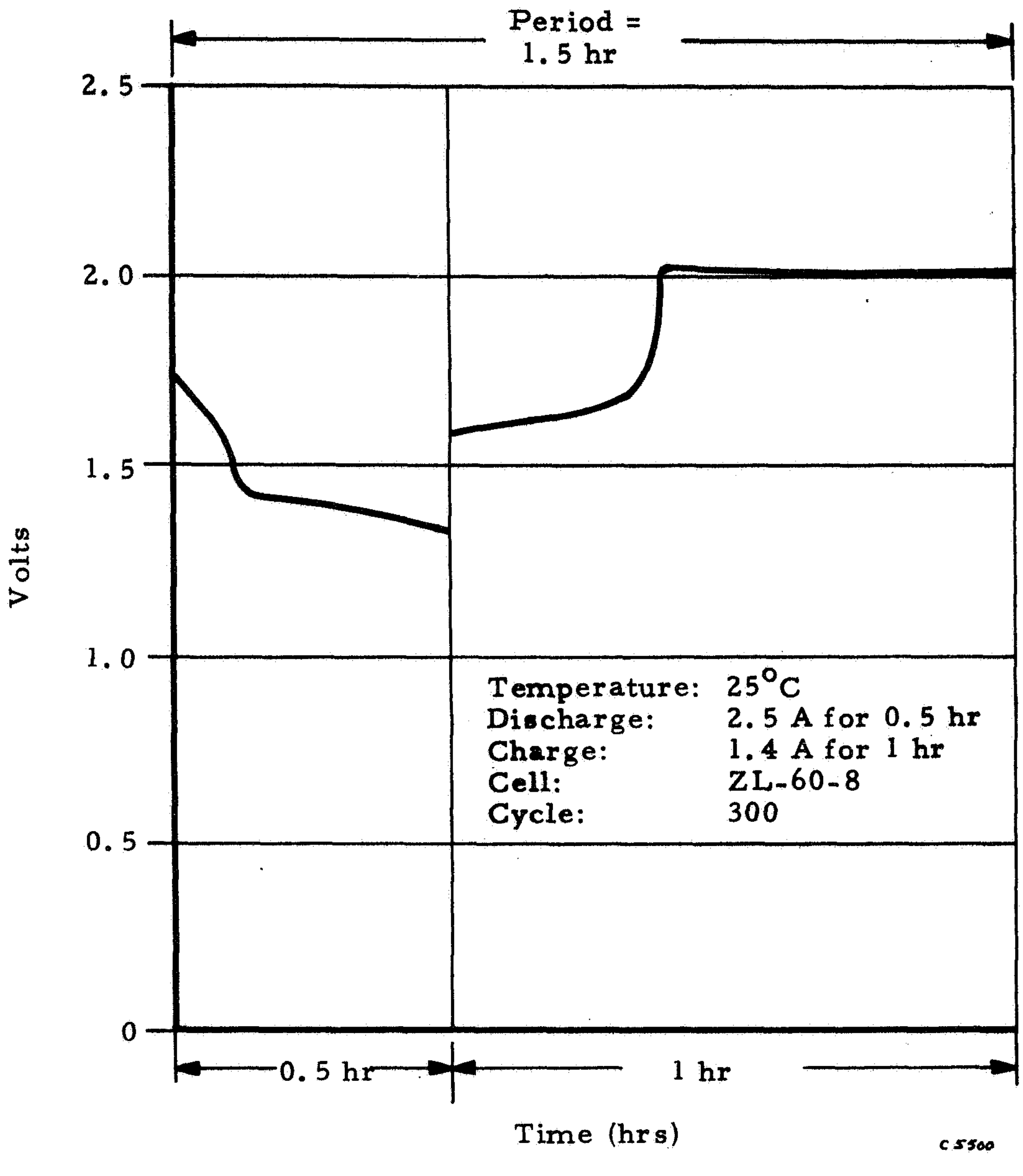


Figure 91. Cycling Curves - Group C

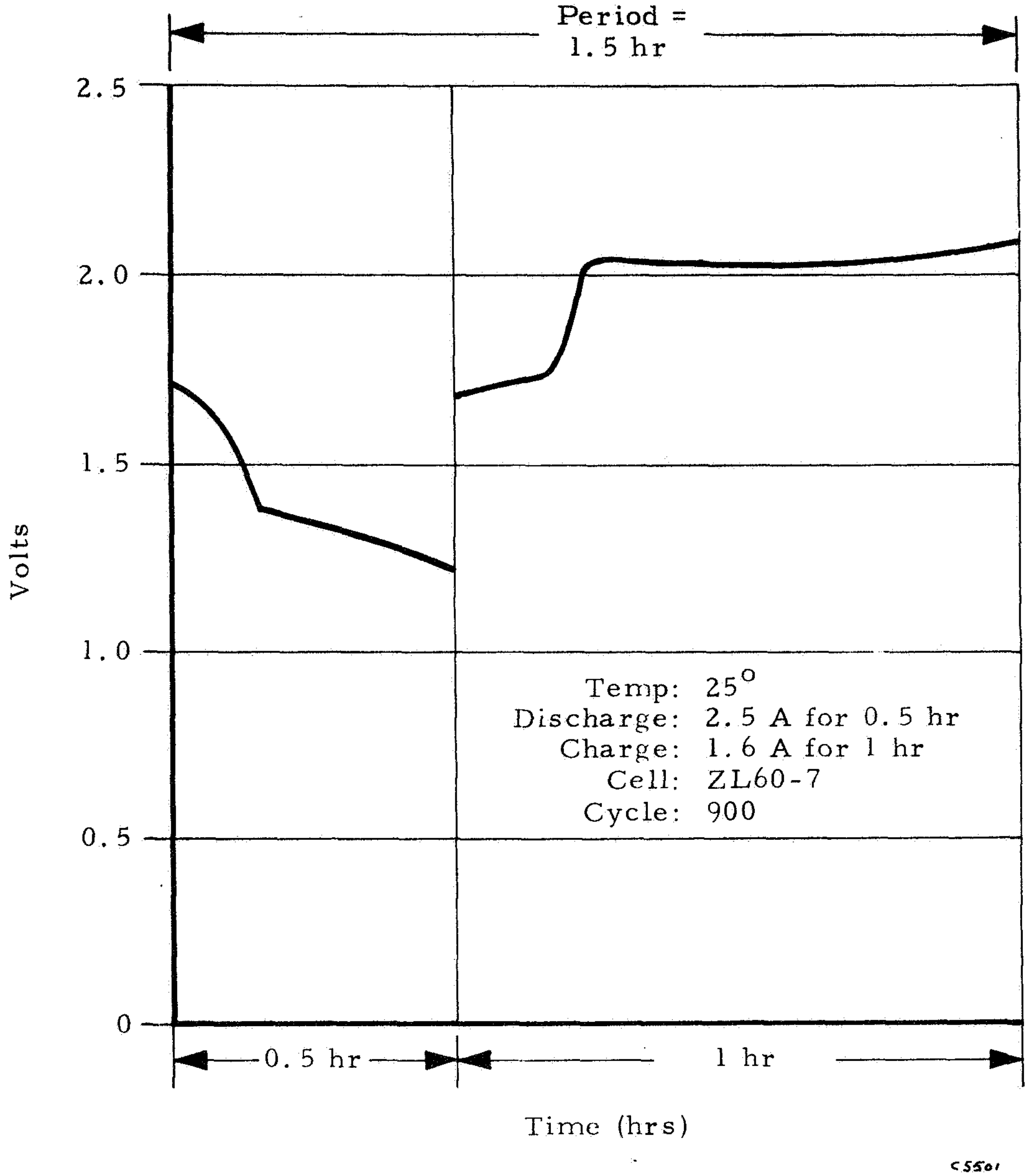


Figure 92. Cycling Curves - Group C

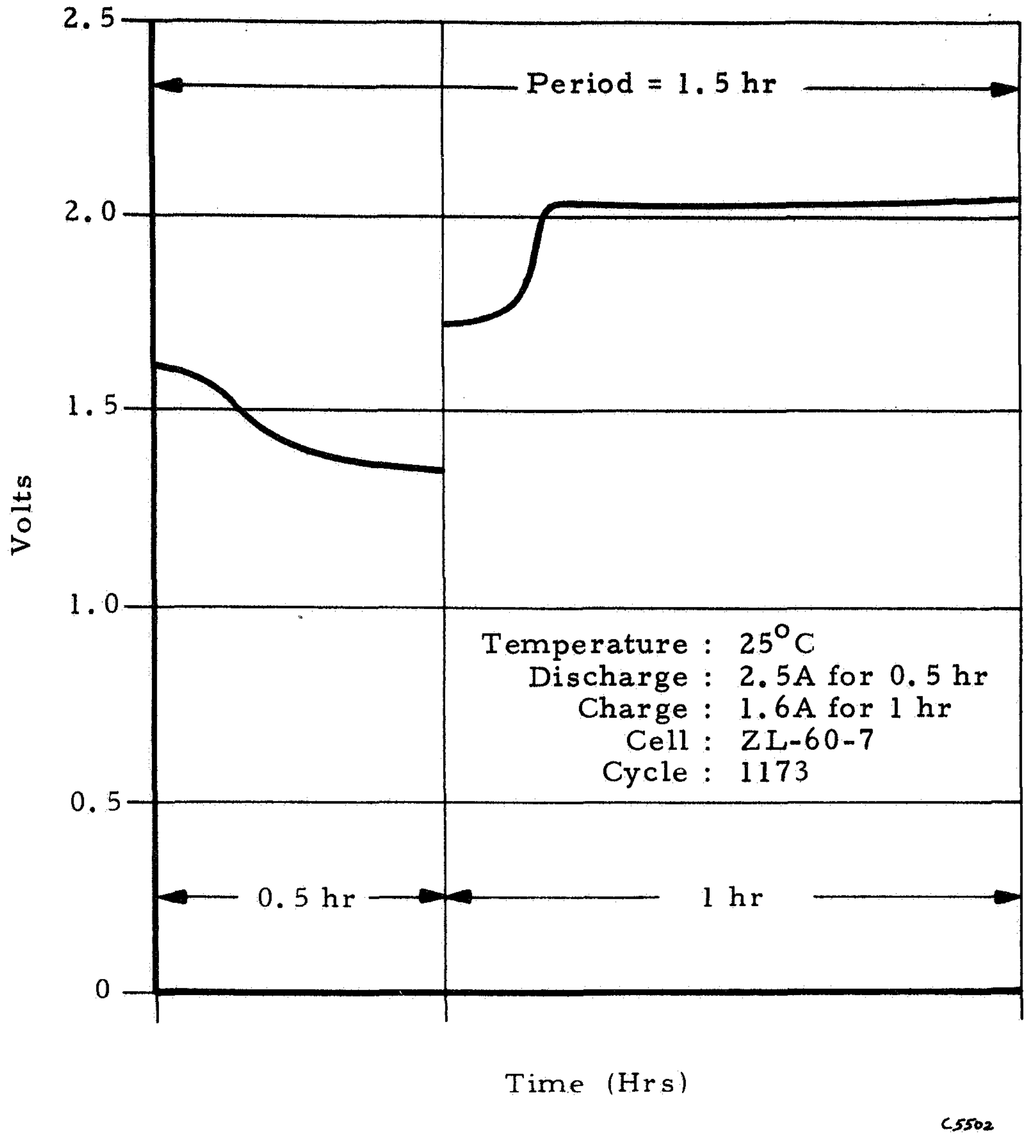


Figure 93. Cycling Curves - Group C

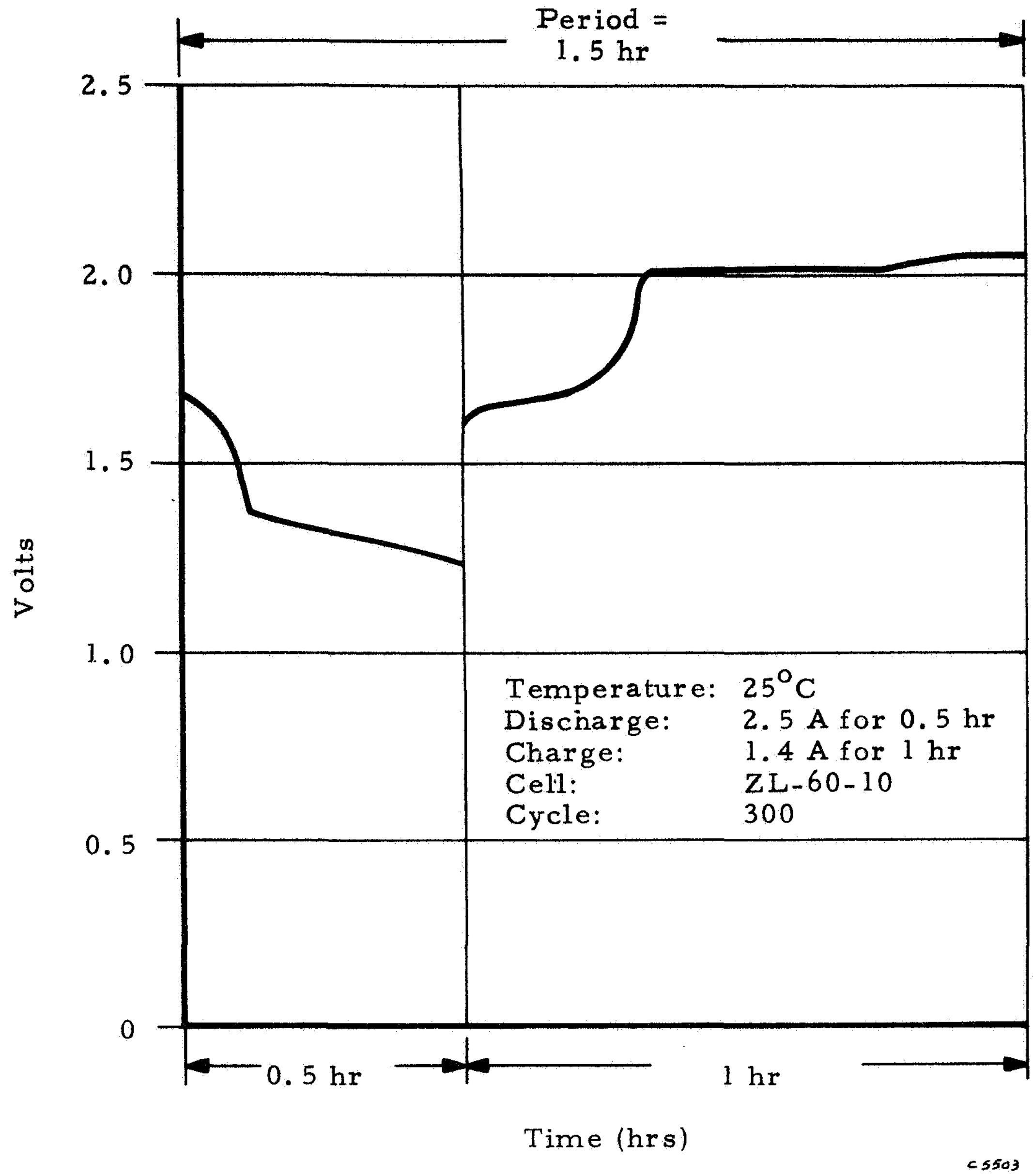


Figure 94. Cycling Curves - Group D

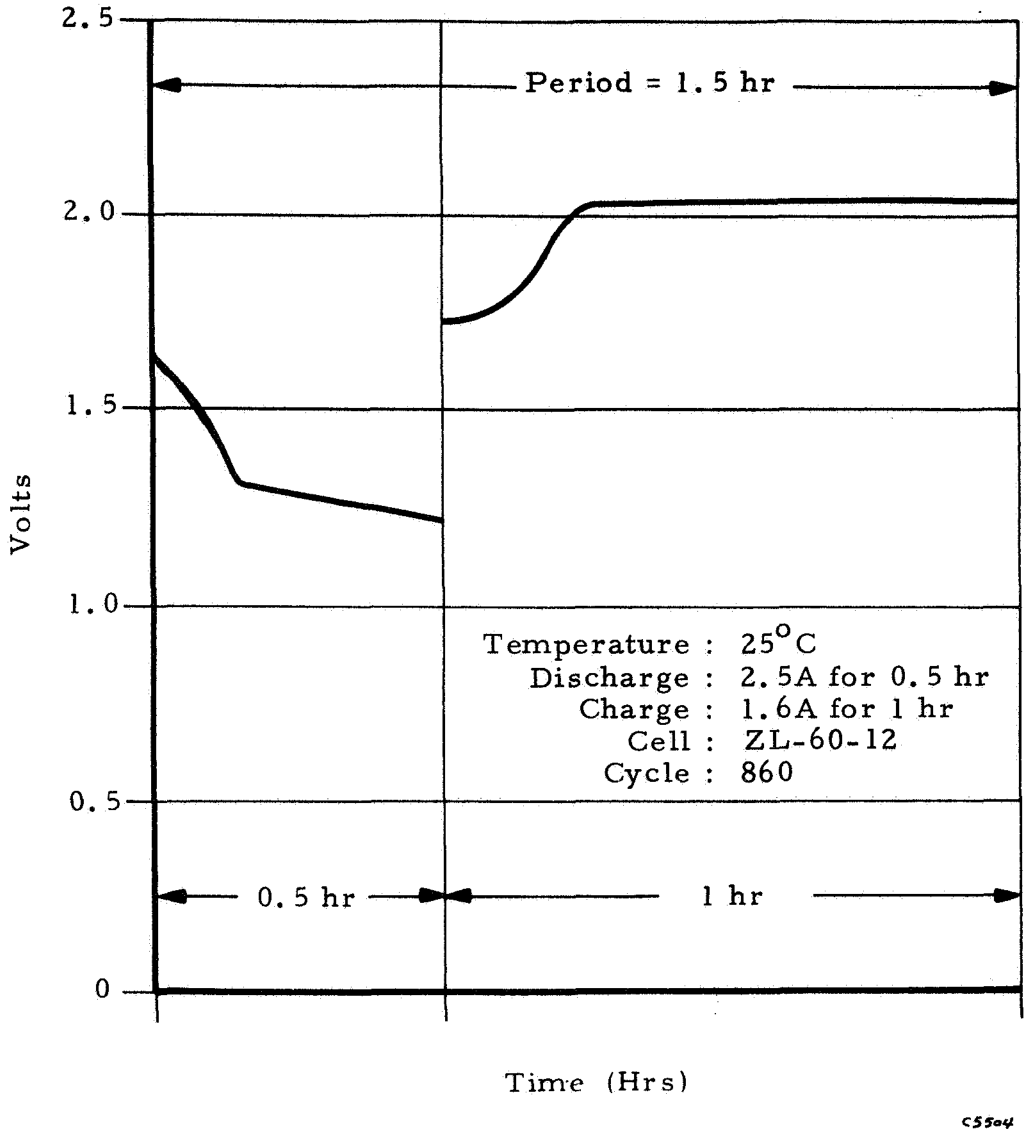


Figure 95. Cycling Curves - Group D

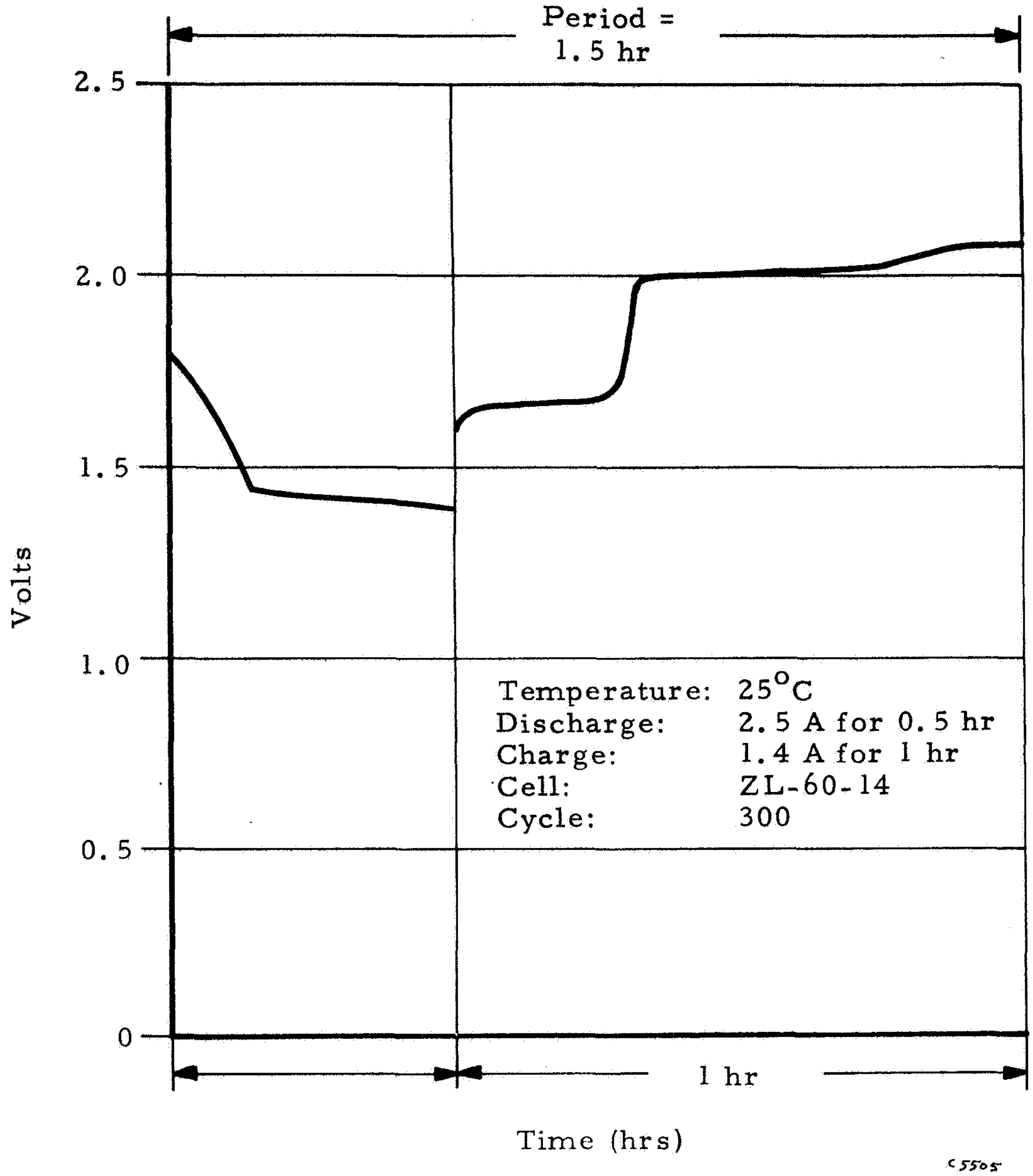


Figure 96. Cycling Curves - Group E

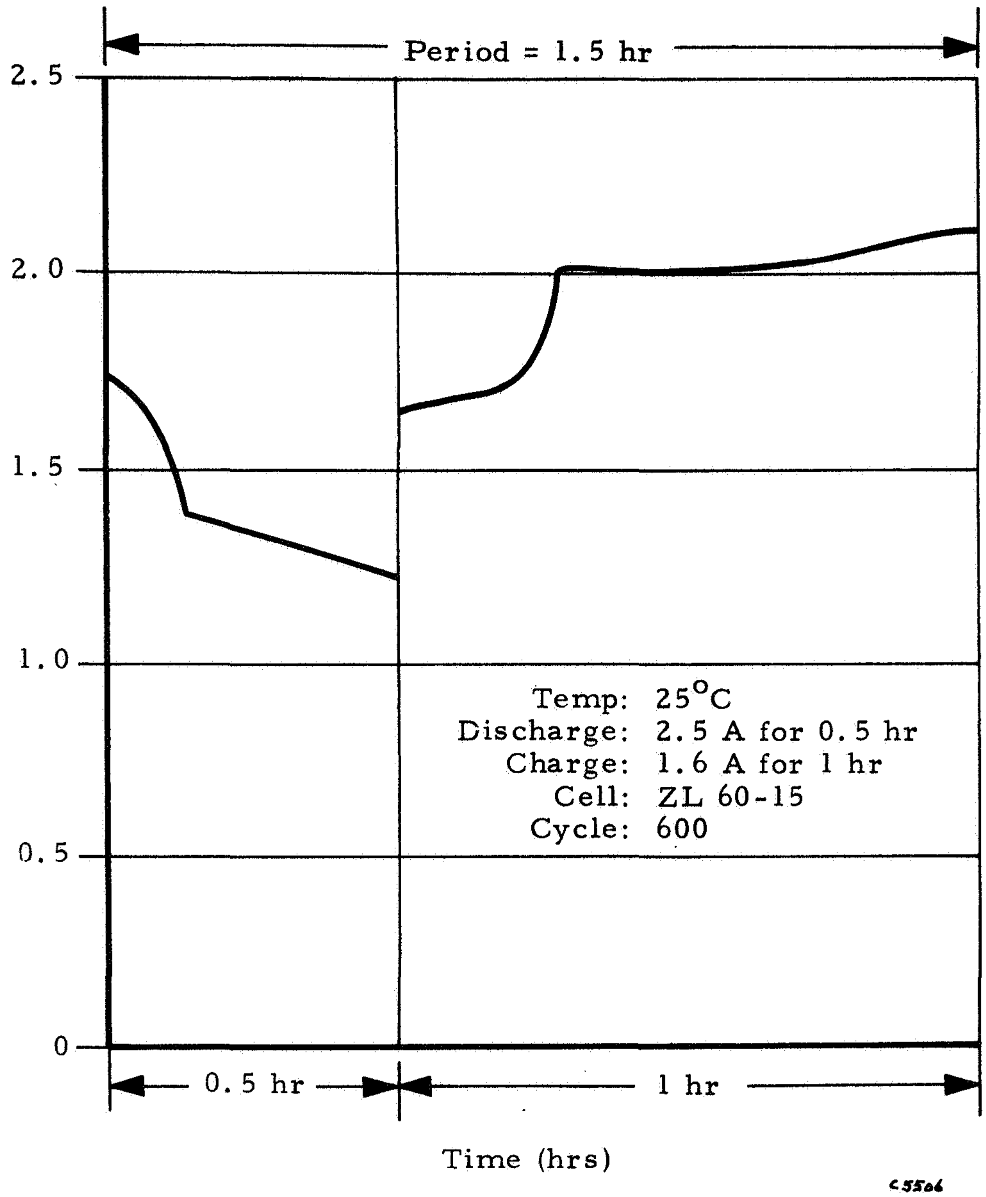


Figure 97. Cycling Curves - Group E

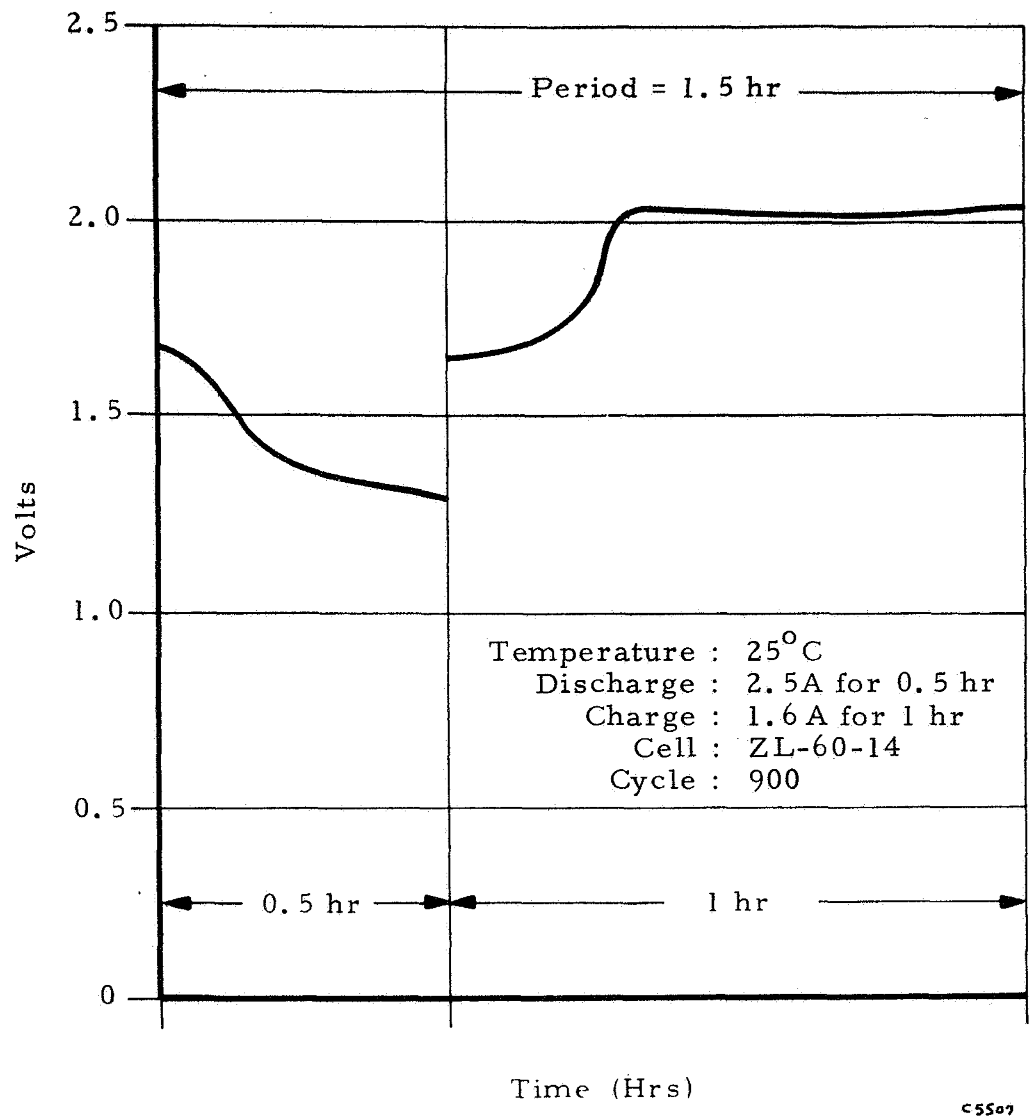


Figure 98. Cycling Curves - Group E

TABLE CII

EFFECT OF CYCLING METHOD
CYCLING DATA
STATUS AT END OF PROGRAM

Group	Cell No.	Cycle
Cells	ZL-62-6	1300
	ZL-62-7	927
	ZL-62-8	1248
	ZL-62-9	946
	ZL-62-10	1010
Battery*	ZL-62-1	817
	ZL-62-2	1025
	ZL-62-3	1025
	ZL-62-4	773
	ZL-62-5	817

* The cells were cycled in series as a battery. After failure of a cell, the cell was removed and the remaining cells were cycled as a battery. Only cells ZL-62-2 and ZL-62-3 were left in series when the test was discontinued.

Table CIII gives their maintenance frequency. Table CIV gives the amount of electrolyte addition required throughout their cycle life. Cycling curves are shown in Figures 99 to 104. Although individual cell cycling shows some beneficial effect on the cycling performance, the wide scatter of data does not allow to draw a firm conclusion. At the end of the program, the cells were removed from test, charged, discharged at 5 A and drained at 0.3 0 A to 1.0 V. The data are presented in Table CV.

TABLE CIII

MAINTENANCE FREQUENCY (ZL-62)

(Cycle Number at Which Maintenance Was Done)

Maintenance Number	Battery	Cells				
	ZL-62-1 to -5	ZL-62-6	ZL-62-7	ZL-62-8	ZL-62-9	ZL-62-10
1	606	1124	530	755	654	560
2	640	1155	595	830	720	640
3	721	1254	661	986	817	683
4	742	1278	737	1015	903	815
5	755	1300	772	1092	946	845
6	767		802	1179		864
7	773		819	1194		911
8	817		863	1228		952
9	883		879	1248		983
10	960		900			999
11	1012		918			1010
12	1025		927			

TABLE CIV

EFFECT OF CYCLING METHOD ELECTROLYTE ADDITION (cm^3)

(Total amount between indicated cycles)

Cycle	Battery (1 to 5)					Cells				
	#1	#2	#3	#4	#5	#6	#7	#8	#9	#10
	0	0	0	0	0	0	0	0	0	0
200										
	0	0	0	0	0	0	0	0	0	0
400										
	3	3	3	3	3	0	3	0	0	3
600										
	2	6	2	6	6	0	6	2	3	0
800										
	2	5	3	0	0	0	3	0	3	6
1000										
		4	0			3		2		
1200										
Total amount up to failure	7	18	8	9	9	3	12	4	6	9
Average per 100 cycles over total life	0.85	1.75	0.78	1.15	1.10	0.23	1.3	0.32	0.78	0.90

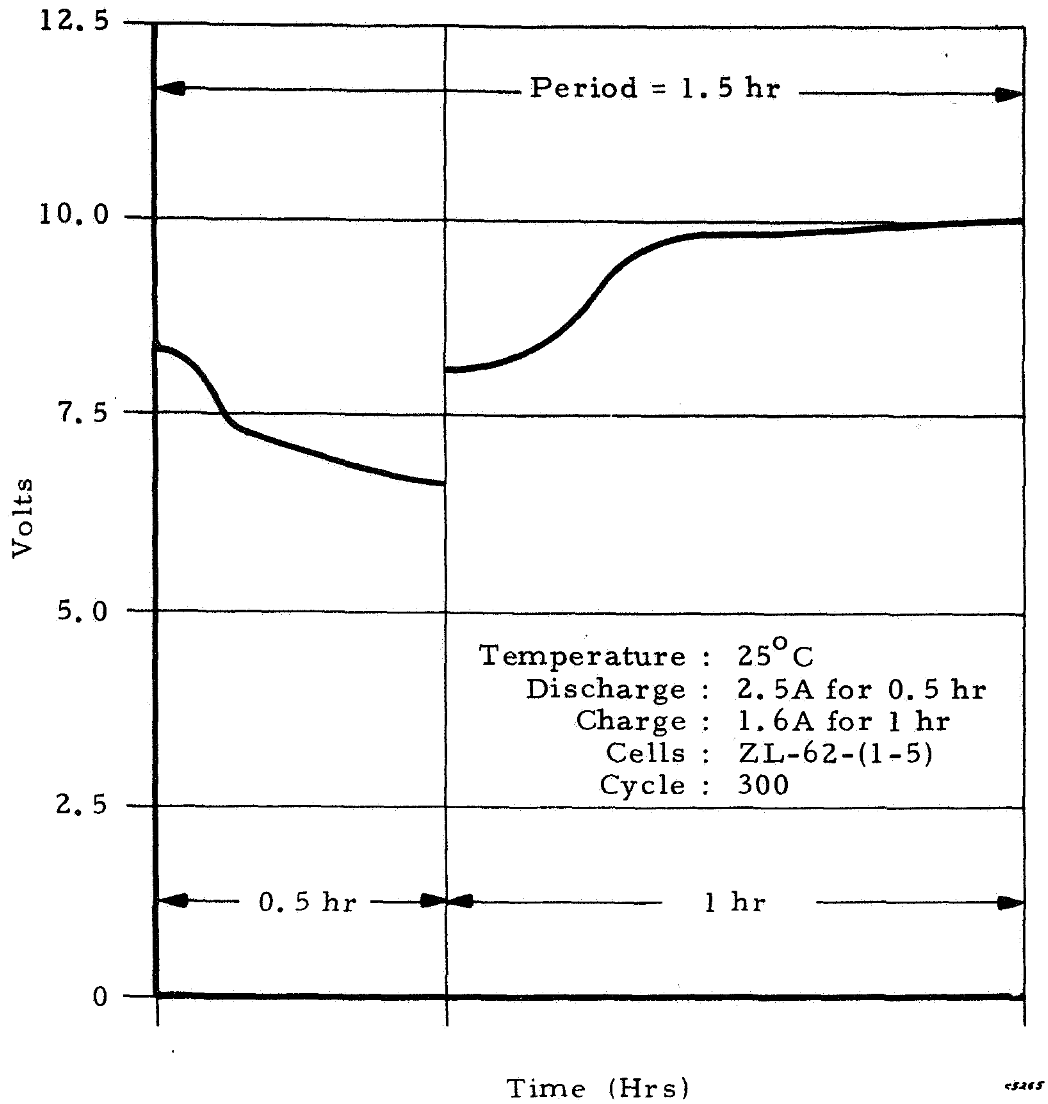


Figure 99. Five-Cell Battery Cycling

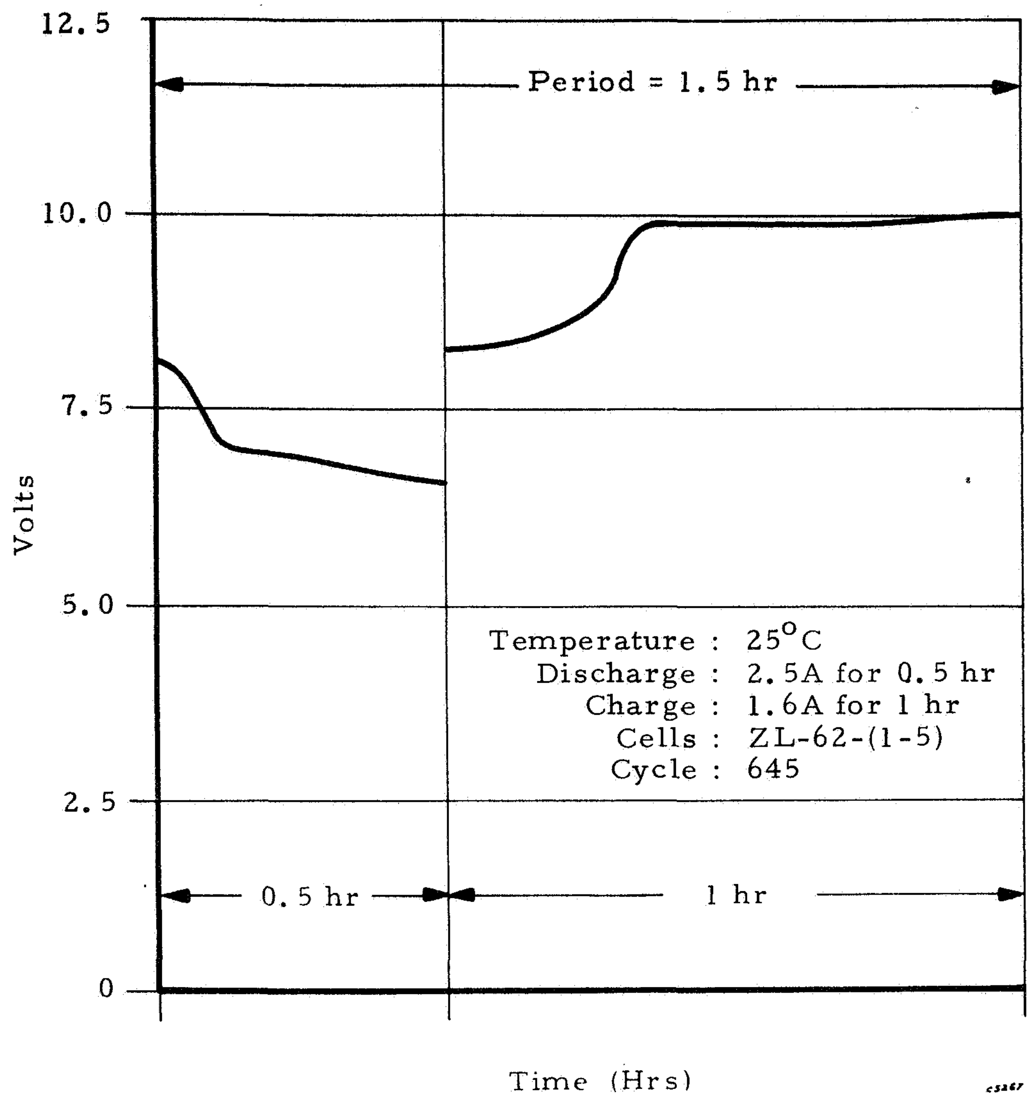


Figure 100. Five-Cell Battery Cycling

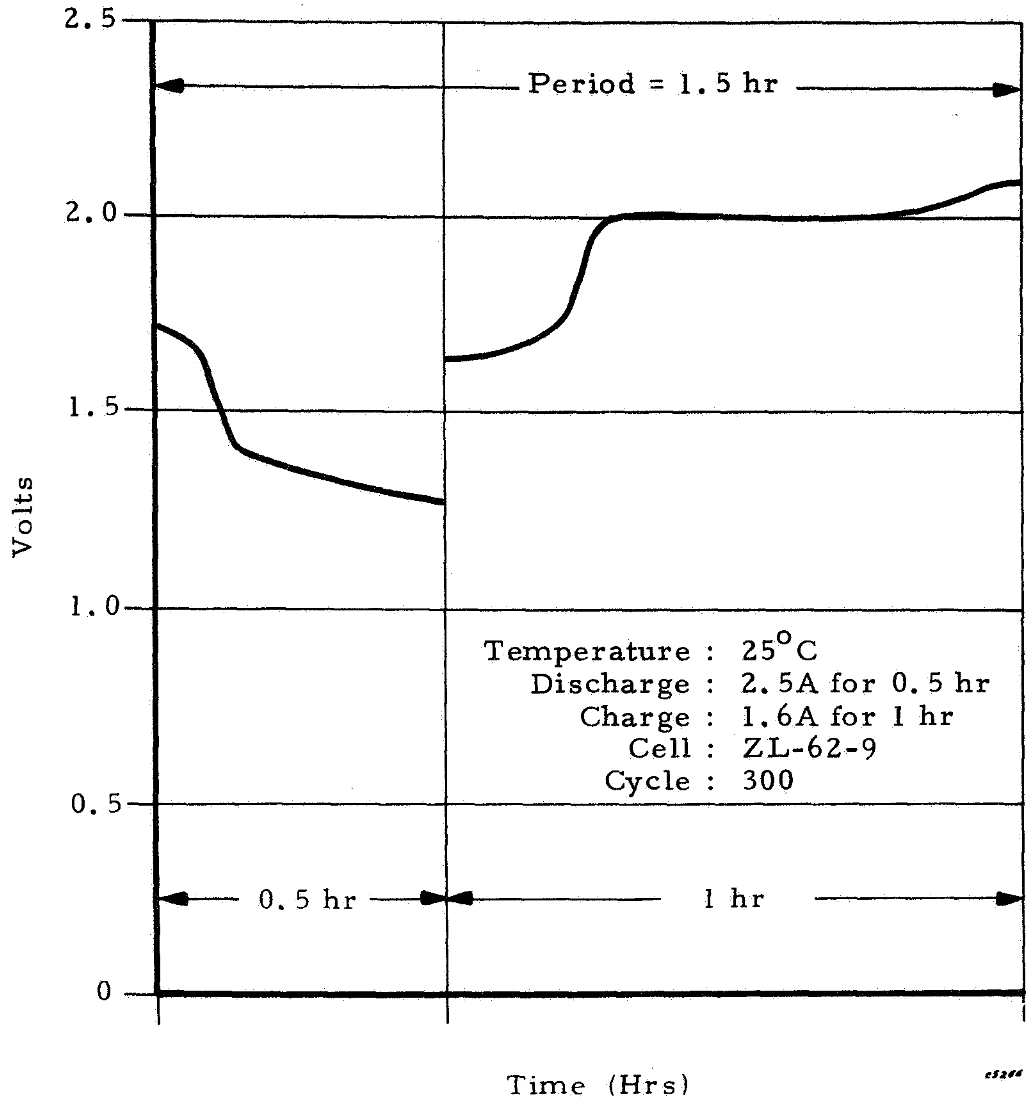


Figure 101. Individual Cell Cycling

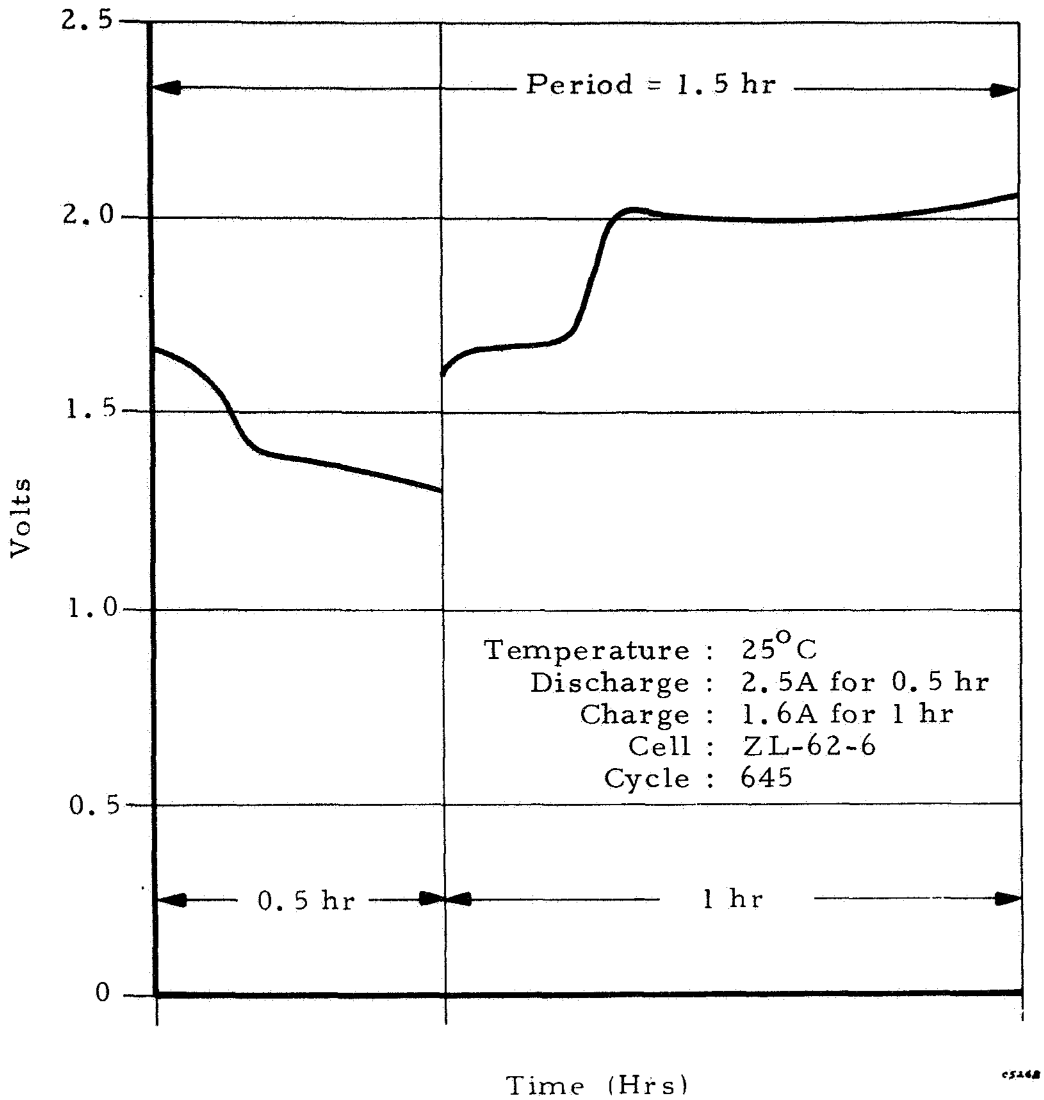


Figure 102. Individual Cell Cycling

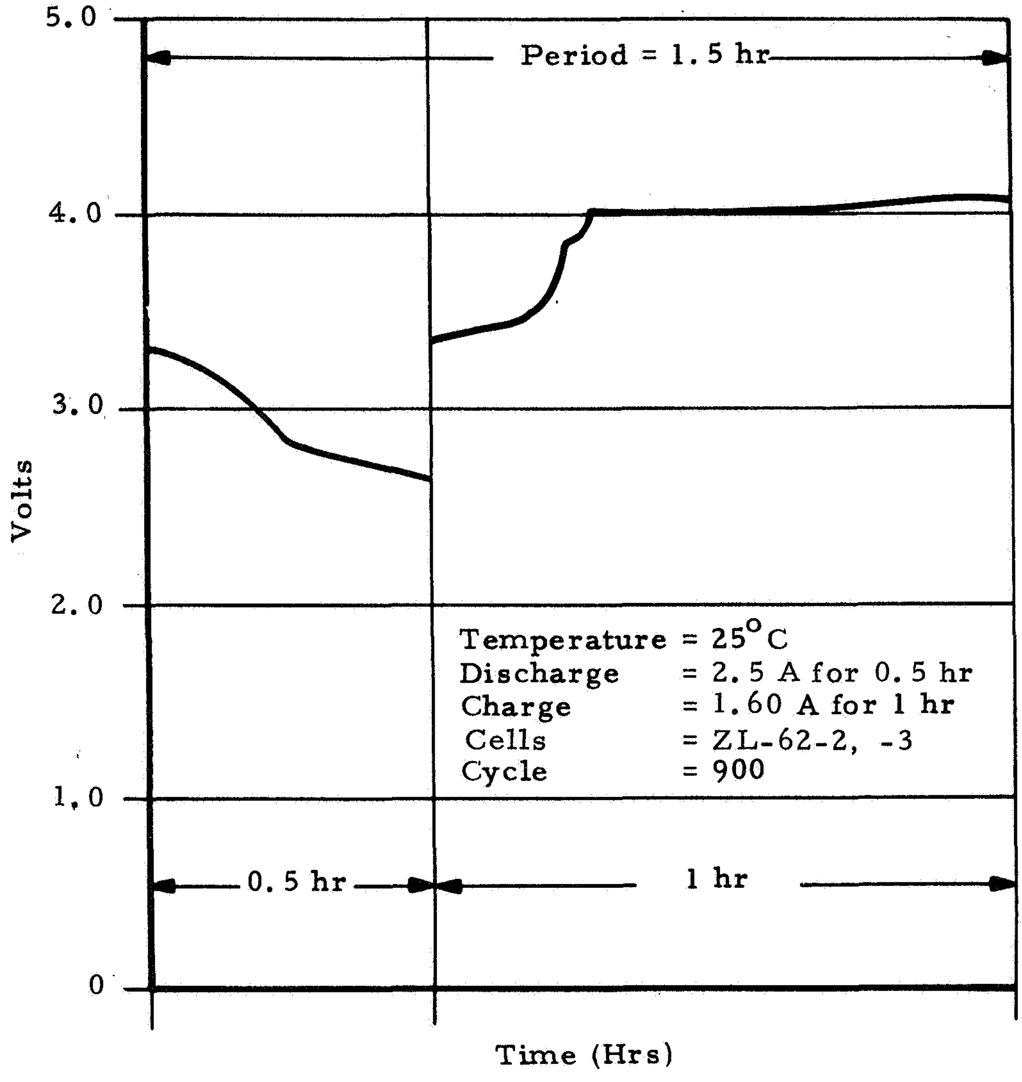


Figure 103. Two-Cell Battery Cycling

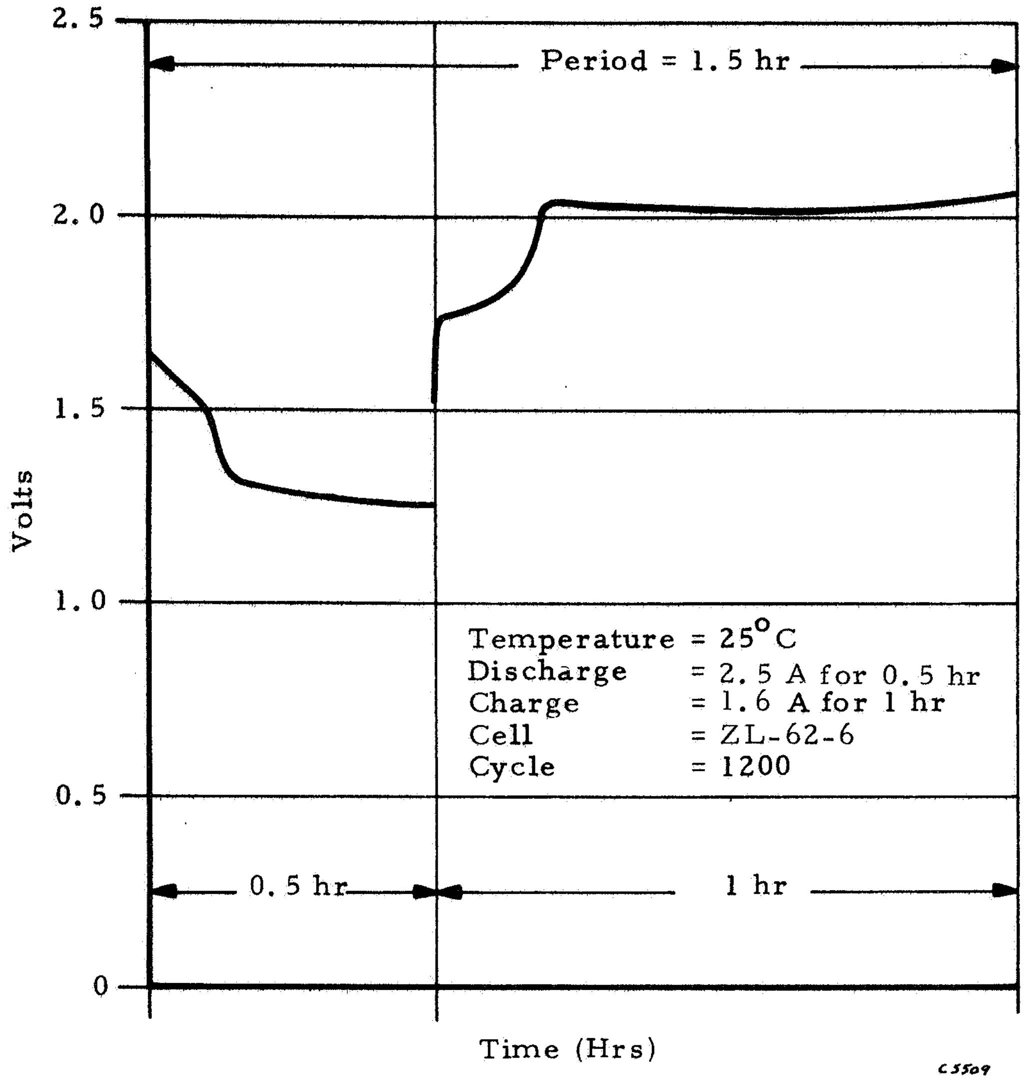


Figure 104. Individual Cell Cycling

TABLE CV

STATE OF CELLS AFTER TERMINATION OF TESTS

Cell No.	OCV after 6 hours	Output at 5 A to 1.0 V	Drain at 0.350 A to 1.0 A	Total Output
ZL-62-2	1.84 V	0 Ah	1.80 Ah	1.80 Ah
ZL-62-3	1.84 V	1.50 Ah	2.50 Ah	4.00 Ah
ZL-62-6	1.85 V	1.75 Ah	0.70 Ah	2.45 Ah
ZL-62-7	1.84 V	3.00 Ah	0.60 Ah	3.60 Ah
ZL-62-8	1.85 V	1.90 Ah	2.60 Ah	4.50 Ah
ZL-62-10	1.84 V	2.00 Ah	0.75 Ah	2.75 Ah

Section 4
SUMMARY OF RESULTS AND CONCLUSIONS

The first year of this program was devoted to refining the cell design by investigation of the critical components of the 5-Ah cells. The silver electrode used a reinforced grid; the zinc electrode deemed acceptable was not modified, although a few tests showed the beneficial effect of PbO additive. The separator used was the same inorganic 3420-09, with stress on the quality control aspect. The case material selected was polysulfone over PPO, only on the basis of transparency. The case-to-cover ultrasonic weld was successful for both materials. The terminal used remained unchanged.

The electrolyte concentration was changed to 40% on the basis of improved wet stand and lower gassing characteristics. However, the 30% should not be completely ruled out, since this concentration minimizes zinc shape erosion.

The study on separator edge seals led to the conclusion of retaining the same material and method as previously developed.

A 40-psig valve was adapted to the cell cover to minimize electrolyte loss and promote gas recombination as shown in gassing studies conducted at three regimes and temperature combinations. Pressurized cells evolved less gas then free-venting cells.

All of these features were incorporated in the final cell design which was submitted to selected tests for final approval. Wet stand tests at room temperature were satisfactory over one year. The environmental tests (shock, acceleration, vibration) showed no detrimental effect on the charged cells. The electrical tests run on a severe regime gave remarkably good results considering the high rates imposed on charge and discharge on a cell intended for a low rate application.

Task III covers the fabrication of 210 cells in 6 different lots to determine reproducibility of manufacture. The cells were divided as follows: 6 lots of 25 cells each were delivered to NASA and 6 lots of 10 cells were submitted to testing in our own laboratory. The cells were tested on 5 different cycling-temperature regimes and on charged wet stand. On the average, the cells are capable of 1500 cycles on the 1.5 hr-cycling period and 20% depth of discharge (based on 7.5 Ah original capacity). After 6-month charged wet stand, they will retain at least 80% of their capacity. With some cycling and 12-month wet life, they still deliver 20% over their rated capacity (5 Ah).

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APPENDIX A

ENVIRONMENTAL TESTS

Appendix A
ENVIRONMENTAL TESTS

Each of ten multiple-plate cells shall be subjected to shock, vibration, and acceleration tests as defined below.

At the conclusion of the environmental tests, the cells shall be examined for defects.

The environmental tests shall be performed in accordance with the following specifications:

SHOCK

The fully charged test cell shall be securely mounted in a test fixture specifically designed to support the cell in each of three perpendicular axes for shock testing. A Hyge 3001 hydraulic impulse shock stand shall be used to perform these tests.

Three shock impulses of 18G's shall be applied to the test cell along each of the three perpendicular axes making a total of 18 shock impulses per cell. Each input pulse waveshape shall be a half-size pulse for a time duration of eight milliseconds. The accelerometer pickups shall be placed directly on the cell case and on the test fixture at the cell mounting interface.

At the conclusion of each shock impulse, the cell shall be examined for cracks, dents or other damage. If there are no evidences of mechanical damage, the cell is acceptable for performing the next shock impulse. If there is evidence of damage, the test shall be terminated and an analysis shall be made to determine the reason for failure. If necessary, design corrections shall be made and additional multiple-plate cells shall be fabricated and tested as specified.

VIBRATION

The test cell shall be securely mounted in a test fixture specifically designed to support the cell in any of the three mutually perpendicular axes for vibration tests. A 6-100 shaker system shall be used to perform these tests. The test cell shall remain electrically inoperative throughout the vibration tests.

A resonant survey test shall be performed to determine the resonant modes of the test fixture and cell. A frequency sweep shall be made from 5 to 16 cps with a displacement amplitude of 0.368 inch and from 16 to 2000 cps at 5 G peak. This frequency sweep shall be performed once along each of the three mutually perpendicular axes. The accelerometer pickup shall be placed at the mounting interface between the vibration fixture and the test cell. During each frequency sweep in each direction, all resonant frequency points shall be recorded. The duration of each frequency sweep in each axis shall be one minute from 5 to 16 cps and ten minutes from 16 to 2000 cps. A resonant frequency test at one-half of each resonant frequency resulting from the resonant frequency sweep noted above, shall be performed for a period of 30 minutes for each resonant frequency.

At the conclusion of each vibration test in each of the three mutually perpendicular axes. the cell shall be examined for cracks, dents or other damage. If there are no evidences of damage, the cell is acceptable for performing the next vibration test. If there is evidence of damage, the procedures as described under Shock tests shall then be followed.

ACCELERATION

The cell shall be securely mounted in a test fixture specifically designed to support the cell in any of the three mutually perpendicular axes for the acceleration tests. A relatively small accelerator shall be used for these tests. The test cell shall remain electrically inoperatige throughout the acceleration tests.

Acceleration tests shall be performed in accordance with the following procedure:

1. A sustained acceleration force of 7 G's shall be applied for five minutes along the longitudinal axis chosen in a direction simulating the lift-off of the transporting space vehicle.
2. A sustained acceleration force of 3 g's shall be applied for five minutes along the longitudinal axis in the opposite direction to that in Item 1 above.
3. A sustained acceleration force of 4.5 G's shall be applied for five minutes in both directions along each of the remaining two mutually perpendicular axes to the longitudinal axis of Items 1 and 2 above.

At the conclusion of each acceleration test specified in Items 1 through 3 above, the cell shall be examined for cracks, dents, or other damage. If there are no evidences of damage, the cell is acceptable for performing the next acceleration test. If there is evidence of damage, the procedures as described for shock tests shall be followed.

APPENDIX B

CELL SPECIFICATIONS OF THE 5 Ah-SILVER-ZINC CELL (DA-5-1N)

Appendix B

CELL SPECIFICATIONS OF THE 5 Ah-SILVER-ZINC CELL (DA-5-1N)

Electrode Pack Configuration: 5 positives, 4 negatives

Positives:

Dimensions: 1.6" x 1.6" x 0.022"
Silver Weight: 4.5 g
Interseparator: Pellon 2505 ML

Negatives:

Dimensions: 1.6" x 1.6" x 0.070"
Interseparator: KT (pressed in electrode)
ZnO Mix: 6.0 g
HgO: 2%

Separators:

Inorganic, rigid, 3420-09
Thickness: 25 mils
Absorption: 10%

Assembly:

Negative electrode sandwiched between two oversized rigid separators forming a compartment sealed on 3 edges and open at the top

Electrolyte: 22 cm^3 of 40% KOH

Case and Cover: Polysulfone P-1700

Cover to Case Seals: Ultrasonic weld and epoxy

Other: 40 psig pressure relief valve

Dimensions (plastic only): 3" h x 2.28" w x 1.04"

Weight (with electrolyte): 230 grams

Original Capacity: 7.5 to 8.0 Ah

APPENDIX C

TRANSVERSE STRENGTH OF INORGANIC SEPARATORS

Appendix C

TRANSVERSE STENGTH OF INORGANIC SEPARATORS

Samples are broken on the transverse testing machine shown in Figure C-1. The modulus of rupture was calculated according to the equation:

$$M = \frac{3Pl}{2bd^2}$$

where,

M = modulus of rupture (psi)

P = breaking load (pounds)

b = breadth (inches)

l = span (inches)

d = thickness (inches)

Tested specimens have the following meacurements:

b = 1/2 inch

l = 1 inch

Formula used:

$$M = \frac{3\,P}{d^2}$$

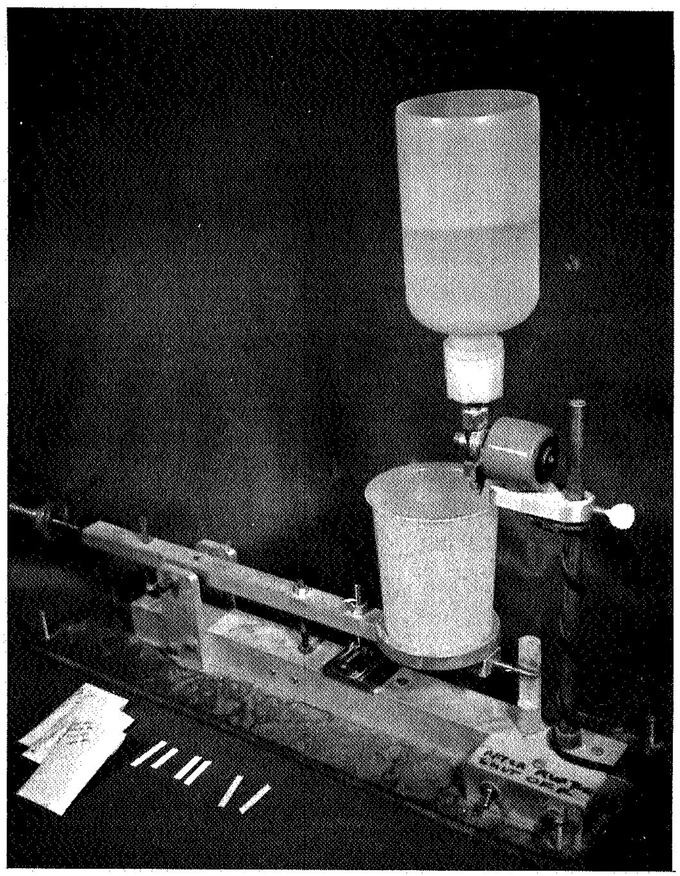

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Figure C-1. Transverse Strength Apparatus

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